STUDIES ON Zn(II) AND Cu(II) IONS ADSORPTION BY RICE HUSKS DIGESTED WITH NITRIC ACID

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by

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Dissertation submitted in partial fulfillment of
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CERTIFICATION OF APPROVAL

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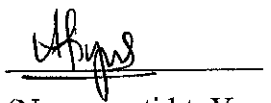
Approved by,



(Normawati bt. Yunus)
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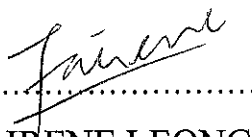
CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



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FAIRENE LEONG CHIN LIN

ABSTRACT

This research studies the effects of different preparation conditions on the yield of activated carbons and also to study the adsorption capacity of activated carbons produced towards Zn(II) ions and Cu(II) ions. In this research, the effects of carbonization time, the weight ratio of nitric acid to rice husks, sample loading size and contact time will be studied while observing the condition of the activated carbons produced from rice husks. This study is significant as Zn(II) and Cu(II) ions removal from industrial and municipal wastewater is vital in view of the many health and environmental hazards caused by heavy metal contaminated water. Since the current methods of Zn(II) and Cu(II) removal with commercial activated carbon are expensive, this study on rice husks based activated carbon is timely as rice husks are readily available and costs less than commercial adsorbents. The methodology employed in this study is to first treat the rice husks with nitric acid at different weight ratios of 1:1, 1.5:1, 2:1 and 2.5:1, before carbonizing at temperature of 500°C varying periods of 1 hour, 2 hours, 3 hours and 4 hours. An FTIR scan was done on one of the samples to determine the functional groups of the activated carbon produced. The activated carbon would then be used as adsorbents to adsorb Zn(II) ions in Zinc(II) Nitrate and Cu(II) ions from Copper(II) Nitrate solutions. The adsorption capacity towards both metal ions will finally be determined using the Atomic Absorption Spectrophotometer (AAS). It was found that the activated carbons are more suitable for the removal of Zn (II) ions as the percentage of removal is higher than that of Cu(II) ions. Carbonization time and weight ratio generally increases the adsorption capacity. However, both parameters have their limits whereby any increment of weight ratio or carbonization time would deteriorate the adsorption capacity. The optimum experimental conditions for maximum removal of Zn(II) ions are 2 hours carbonization time with nitric acid to rice husks weight ratio of 1:1, loading size of 0.4 g and contact time of 1hour. As for Cu(II) ions removal, the optimum experimental conditions are 4 hours carbonization, 2.5:1 weight ratio ,0.4g loading size and 4 hours contact time.

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ABBREVIATIONS AND NOMENCLATURE

b	Equilibrium constant related to Henry's constant
c	Equilibrium concentration, kg adsorbate/ m^3 fluid
K	Henry's constant
$1/n$	Empirical constant, function of temperature
q	Quantity of adsorbed material, kg adsorbate/kg adsorbent
qo	Empirical constant, kg adsorbate/kg adsorbent
t	Toth parameter

CHAPTER 1
INTRODUCTION

1.1 Background of study

In today's growing industrial age, the rate of water contamination has increased a great deal. Rivers or streams situated near industrial sites have high content of free metal ions, particularly that of heavy metals. Heavy metals are metals with densities exceeding 5 kg/dm^3. They are divided into two categories namely metals with undesired effect (Fe, Mn, Cu, Zn, Co, Ba, Ag) and metals giving a negative toxic effect (As, Cd, Cr, Hg, Ni, Pb, Sb, Se) (SOILWATCH, 1998).

Available technologies for wastewater treatment are precipitation, coagulation or floatation, sedimentation, electrochemical techniques, ion exchange and biological process. However, earlier research has shown that adsorption process is highly effective in removing trace amounts of metal ions such as Zn(II) and Cu(II). Due to the costliness of commercially available adsorbents, there is a need to produce cheaper activated carbon from agricultural wastes such as coconut shells, bones, tree leaves, sugar cane bagasse, and rice husks (Castro et. al., 2000).

Various agricultural wastes are being studied extensively as a potential raw material as they are available in abundance, renewable and cheaper to produce. In addition, since these materials are non toxic, the safety of use in wastewater and water treatment is guaranteed. In Malaysia, rice husks have a high potential to be used as adsorbent as they are readily available nationwide throughout the year. Naturally, rice husks are carbonaceous materials that possess a large surface area. Hence this provides a solid

reason to carry out studies on the adsorption process of Zn(II) and Cu(II) using activated carbon produced from rice husks digested in nitric acid.

1.2 Problem statement

Zinc(II) and Copper(II) are two common heavy metal ions found in industrial wastewater. Rivers and streams are depositing zinc and copper polluted sludge on their banks while the acidity of the water may have also increased. This leads to contamination of waterways, and contamination of groundwater and soil. Fish that live in contaminated waterways allow zinc into the food chain as the zinc ions enter their bodies and accumulate. Consumption of contaminated fish, water and crops grown on contaminated soil can is detrimental to the health of humans and animals alike. Exposure to excessive zinc ions causes stomach cramps, skin irritation, vomiting, and copper deficiency while excessive consumption of copper ions could cause gastrointestinal problems. (Pizarro et. al., 2001).

Since the current methods of zinc and copper metal ions removal are costly, a cheaper option of adsorption process must be formulated, replacing expensive commercial activated carbon with those produced from agricultural waste such as rice husks. Rice husks are readily available from rice mills and paddy fields in Malaysia. Apart from being used for boilers in industries, the rice husks are normally burnt in open burning. This act could further contribute to the pollution of air. Hence, using the rice husks as raw material for activated carbon not only reduces the cost of Zn(II) and Cu(II) ion removal but also reduces the incidences of open burning.

This significance of this project is indeed apparent as there are limited established researches done on the adsorption of Zn(II) ions and Cu(II) by activated carbon produced from rice husks digested in nitric acid. Most available research work revolves around "heavier" metal adsorption such as mercury ions removal by rice husks or usage of other agricultural wastes. Hence, carrying out this study investigate the optimum conditions for the rice husk activated carbons is indeed timely. This study will also

serve as a confirmation or comparison of the results obtained by the former Final Year Research Project students, Ng Joon Leong and Teo Ching Synn. It is hoped that this research project can contribute significantly to the development of rice husk based activated carbons in the future.

1.3 Objectives and Scope of Study

The main objective of this study is to investigate the feasibility of utilizing the rice husks as a raw material to produce activated carbon that can adsorb Zn (II) and Cu (II) ions from aqueous solutions. This study also aims to determine the optimum conditions for the rice husk activated carbons. The scope of the study is focused on the following objectives:

1.3.1 To study the effect of carbonization time on the adsorption of Zn(II) and Cu(II).

Samples carbonized for 1 hour, 2 hours, 3 hours and 4 hours are used to adsorb Zn(II) ions and Cu(II) ions from $Zn(NO_3)_2$ and $Cu(NO_3)_2$ solutions, respectively.

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1.3.2 To study the effect of Nitric Acid to Rice Husks Weight Ratio on the Adsorption Capacity of Zn(II) and Cu(II) ions.

Rice husks are digested with nitric acid based on varied weight ratios of nitric acid to rice husks. The ratios tested are 1.0:1.0, 1.5:1.0, 2.0:1.0 and 2.5: 1.0

1.3.3 To study the adsorption capacity of activated carbon produced for adsorption of Zn(II) ions and Cu(II) ions.

Concentration of Zn(II) ions and Cu(II) ions adsorbed by rice husks are examined using the Atomic Absorption Spectrophotometer.

1.3.4 To observe the condition of the rice husks as they are processed into activated carbon.

Changes in physical appearance and texture are noted down as the rice husks are processed.

CHAPTER 2
LITERATURE REVIEW AND THEORY

2.1 Adsorption Process and Wastewater Treatment

Adsorption is a process of accumulating substances from a solution on a suitable interface. It is a mass transfer operation in that a constituent in the liquid phase is transferred to the solid phase. The adsorbate is the substances that are being removed from the liquid phase at the interface. The adsorbent is the solid, liquid or gas onto which the adsorbate accumulates. In this case, only liquid-solid interface shall be discussed (Metcalf et. al., 2004).

Adsorption from solution onto a solid occurs as a result of one or both of two characteristic properties for a given solvent-adsorbate-adsorbent system. The primary driving force relate to the solvophobic or lyphobic character of the adsorbate or sorbent. For the adsorbates, the parameters are the concentration, molecular weight, size, structure, polarity, steric form or configuration and the nature of competitive adsorbates. The parameters for adsorbents are surface area, the physicochemical nature of the surface, the availability of that surface to adsorbate ions and the physical size and form of the adsorbent particles. System parameters such as temperature and pH can also influence adsorption.

Adsorption process has not been used extensively in wastewater treatment but demands for better quality of treated wastewater effluent have lead to extensive examination of adsorption on activated carbons Activated carbon wastewater treatment is considered polishing process for water that has already been treated biologically (Metcalf et. al., 2004).

There are a few liquid adsorption isotherms classified into four categories (denoted as the S, L, H and C types) with subdivisions of each type (Do, 1998).

The various isotherm expressions for liquid phase adsorption according to Geankopolis, (1993) and Do, (1998) are:

Linear isotherm $q = Kc$ [Eq. 2.1]

Langmuir isotherm $q = \frac{q_o c}{K + c}$ [Eq. 2.2]

Freundlich isotherm $q = Kc^n$ [Eq. 2.3]

Toth isotherm $\frac{q}{q_o} = \frac{c}{(b' + c')^{1/t}}$ [Eq. 2.4]

Langmuir-Freundlich isotherm $\frac{q}{q_o} = \frac{bc^{1/n}}{1 + bc^{1/n}}$ [Eq. 2.5]

2.2 Activated carbons

Many adsorbents have been developed for a wide range of separations. Normally, adsorbent has various shapes, including small pellets, beads, or granules. A particle of adsorbent has a very porous structure with many fine pores and pore volume up to 50% of the total volume. The adsorption often occurs as a monolayer on the surface of the fine pores. There are a number of activated carbons namely powdered activated carbon (PAC) and granulated activated carbon (GAC). Figure 1 shows the general flow diagram for manufacturing of activated carbons which will be described in the entailing paragraph.

Activated carbon is the universal standard means for purification and removal of trace organic contaminants from liquid and vapor streams. The raw material for activated carbon are carbonaceous matters such as wood, peat, coals, petroleum coke, bones, coconut shell, and fruit nuts. These materials are charred by heating the base material to

a red heat (below 700°C) to drive off hydrocarbons. This is called the carbonization process which is also the pyrolysis process. The char is then "activated" by exposing it to oxidizing gases such as steam and CO_2 at temperature ranging 800°C to 900°C. Activation can also be done by using zinc chloride or phosphoric acid as these chemicals have catalytic effect on pyrolytic condensation of carbohydrates. In this process, micropores is formed when carbonization takes place around fine crystals of inorganic salt and washing of the salt or acid after carbonization produces micropores which are larger than those formed by gas activation.

In water treatment, activated carbon has been widely used because of large surface area (from 500 up to 1500 m^2/g) due to high internal porosity with average pore diameters of 10 to 60 Angstrom. Activated carbons used in this project are produced from rice husks instead of using the commercial activated carbon (Bansal et. al., 1988).

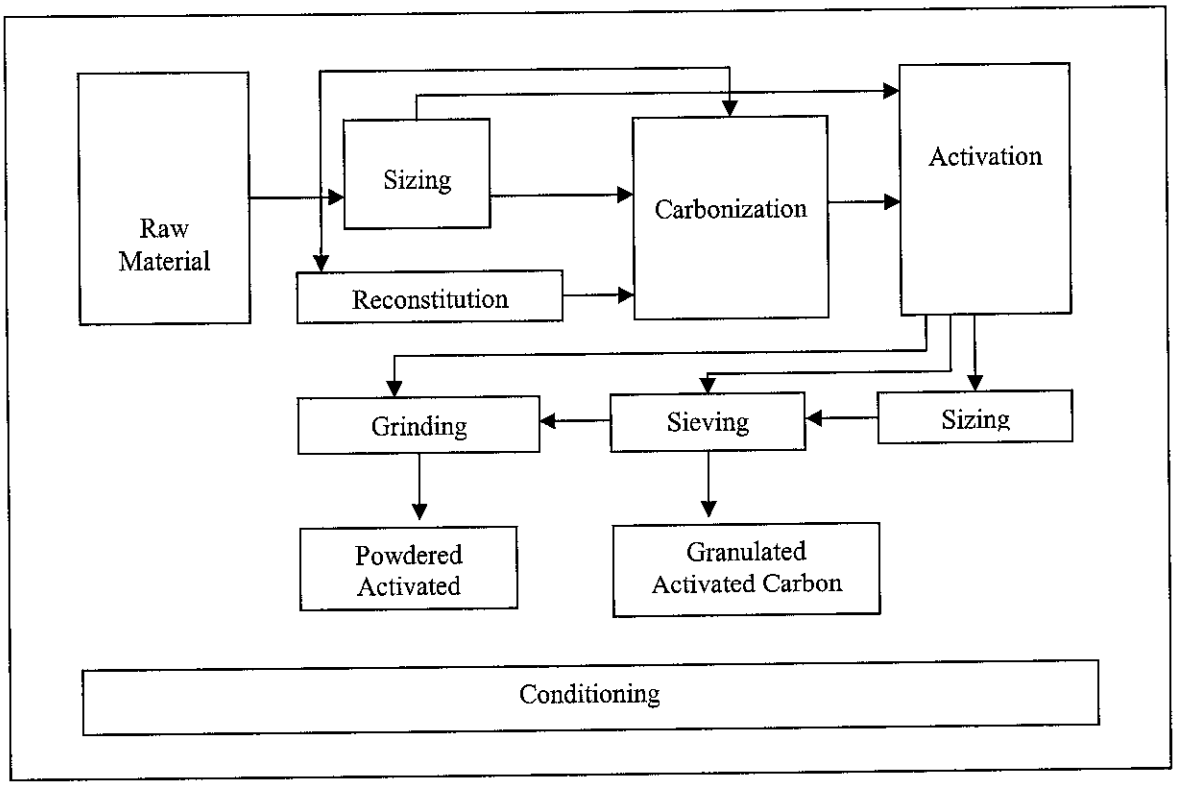

Figure 1: General flow diagram for manufacturing of activated carbon

2.3 Properties of Rice Husks

Rice husks mainly contain organic materials i.e. cellulose, hemicellulose and lignin and hydrated silica. At present, rice husks are the raw materials for the production of silicon-based materials, including silicon carbide, silica, silicon nitride, silicon tetrachloride, pure silicon and zeolite (Sun et.al, 2001). Below is the analysis of rice husks carried out by Luyi Sun and Kecheng Gong in their research entitled "Silicon based materials from Rice Husks and Their Applications":

Table 1: Composition of Rice Husks

Component	SiO_2	K_2O	Na_2O	CaO	MgO	Fe_2O_3	P_2O_5	Cl
Content (%)	86.9-97.3	0.58-2.5	0-1.75	0.2-1.5	0.12-1.96	trace	0.2-2.85	trace

The contents of rice husks were also analyzed through proximate analysis, ultimate analysis and component analysis. The following are the results from an analysis done by Paul J. Williams (Williams et. al., 2000).

Table 2: Proximate, Ultimate and Component Analysis of Rice Husks

Proximate Analysis (Wt %)		Ultimate Analysis (Wt %)		Component Analysis (Wt %)	
				Cellulose	34.4
Volatiles	59.5	Carbon	44.6	Hemicellulose	29.3
Moisture	7.9	Hydrogen	5.6	Lignin	19.2
Ash	17.1	Oxygen	49.3	Ash	17.1

2.3.1 Rice Husks and Activated Carbons

A number of studies on converting rice husks to activated carbon have been reported. In most cases, rice husks were pyrolysed or carbonized in an inert atmosphere (nitrogen or argon) in order to remove volatile organic constituents leaving behind highly porous carbonaceous materials. Normally the materials have to be activated by chemicals

before pyrolysis of rice husks in order to obtain relatively good adsorption capacities. Activation and pyrolysis of rice husks with $ZnCl_2$, H_3PO_4 or CO_2 have been used for adsorption of methylene blue and benzene. It was reported that Cr(IV) could easily be removed from solution by rice husks. The husks were activated with $ZnCl_2$ followed by pyrolysis at different temperatures. Other activating agents such as $ZnCl_2$ and HCl, $ZnCl_2$ and HNO_3 and K_2CO_3 were also used. Digested residue of rice husks consists of a highly porous structure which has a high potential as an effective adsorbent. The property was evaluated with the adsorption of Zn(II) as adsorbents since the cations are common industrial wastes (Rahman et. al., 2000).

2.3.2 Characterization of Rice husks Digested by Acid

Rahman et.al. in their research concluded that hemicellullose and lignin are readily digested by HNO_3 as evident from the significant reduction of their contents after 4 hours digestion. It was observed that the reaction was exothermic with overall mass loss of 68% to 74%. Samples digested for 10 hours showed a reduction of hemicellulose and lignin which consist of 3% and 1.09 %, respectively. However, after 12 hours digestion, the lignin and hemicellulose reduced to trace amounts and remained unchanged. Cellulose was digested at a slower rate as compared to hemicellulose and lignin. About 23% cellulose remained even after 12 hours digestion due to its crystalline structure. Silica was found to be almost insoluble in HNO_3. Hence the increase of silica content with time of digestion was due to the reduction of weight of other components from the overall weight.

Physically, the particle size of husks decreases with increase of digestion time. The color of the original rice husks change from dark brown to yellowish light brown and white. After 12 hours of digestion, the husks were reduced to white powdery form which is actually the skeleton containing 26% cellulosic material and 74% silica with traces of lignin. Table 1 shows the results of the composition of organic constituents in the digested rice husks samples prepared by Rahman et.al., (2000). RH represents raw

rice husks, DH4, DH6, DH8, DH10 and DH12 represent rice husks digested at room temperature for 4, 6, 8, 10 and 12 hours, respectively.

Table 3: Composition of organic constituents in the digested rice husks as compared to the raw rice husks

Composition (%)	Sample					
	RH	**DH4**	**DH6**	**DH8**	**DH10**	**DH12**
Silica	16.1	63.0	68.0	72.0	74.0	74.0
Cellulose	35.5	24.0	23.2	23.2	23.0	22.8
Hemicellulose	22.3	7.1	5.2	3.5	3.0	3.1
Lignin	13.6	6.1	4.2	2.4	1.9	0.2
Solubles and moisture	12.5	-	-	-	-	-
Total	100.0	100.2	100.6	101.9	101.9	100.1

Another observation is the porosity. The porosity of rice husks increase with the increase of digestion time. By pyrolyzing digested rice husks at 700°C, for 1 hour, a material with surface area larger than 30% was produced. This indicates that the thermal composition of cellulosic material will form more pores (Rahman et. al., 2000).

2.4 Effects of Nitric Acid Treatment

Liquid-phase oxidation with HNO_3 greatly enhances the adsorption capacity of all carbons (up to three times). Metal uptake by activated carbon is promoted relative to that of the untreated char. Metal ion uptake has been demonstrated to be a function of polar or acidic surface groups on the carbon surface. It is favored by the presence of oxygen – containing functional groups and the electrostatic attraction of the metal for these sites. The existence of surface oxygen functionalities was detected following the carbon surface modification with nitric acid. These acidic groups lead to a low point of zero charge values and greatly increase the surface charge. This makes metal ions more accessible to the inner pores. Surface oxygen groups enhance the hydrophilic character

of the carbon surface, thus promoting metal adsorption and pore diffusion of the aqueous and hydrated metal ions (El Hendawy, 2003).

2.5 Carbonization

Plant biomass pyrolysis is accompanied by two main groups of chemical transformations namely thermal depolymerization to lower mass products and the dehydration with subsequent condensation of macromolecules. The combination of these processes results in the formation of disordered aromatic layers at 300°C-700°C. Within this temperature interval, no significant variation of surface area and micropore volume was observed, but a non-monoatomic change of micropore sizes was detected. Higher temperatures of carbonization (>700°C) promote the formation of a more ordered structure of carbonaceous material with a simultaneous increase in pore sizes.

The texture of the carbon products is dependent on the rate of heating during lignin carbonization. High rates of heating decrease the BET surface area and micropore volume. However the average size of micropores increases and this promotes mesoporosity development. At these higher heating rates the reactions of lignin depolymerization dominate during carbonization up to 700°C. Lower heating rates promote condensation reactions which favors the development of a microporous structure with minimum pore size (Baklanova et. al., 2003).

CHAPTER 3

METHODOLOGY AND PROJECT WORK

3.1 Generalized Procedure for Activation Carbon Preparation

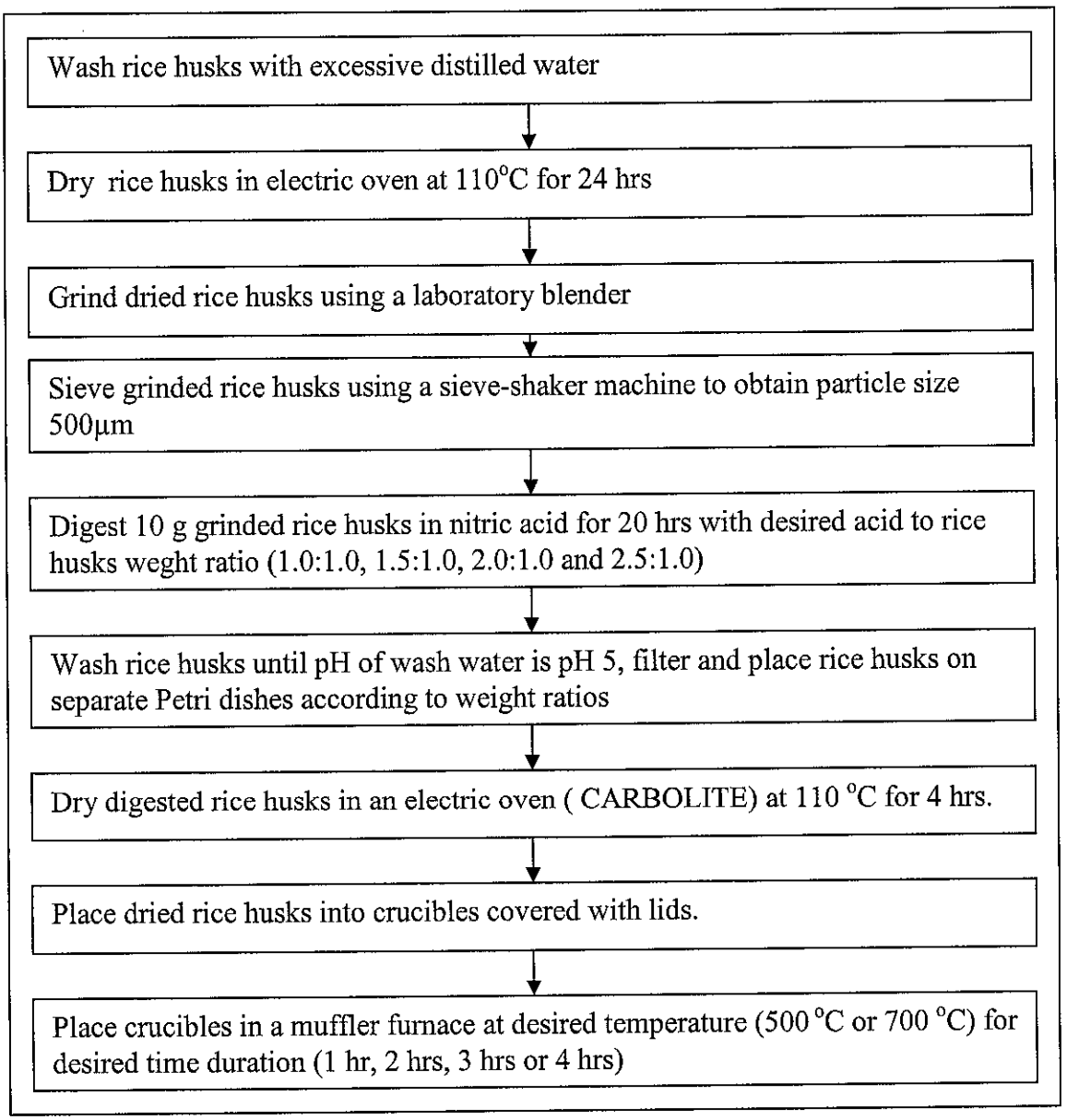

Figure 2: General flow diagram for activation carbon preparation from rice husks

Figure 2 shows the steps taken in producing the activated carbons based on rice husks. Prior to the first stage, a large amount of rice husks was obtained from the Tan Teng Yu Rice Mill Sdn. Bhd, Lumut. For the first four stages, about 500 g of rice husks was processed to cater for the 16 samples that will be needed in the adsorption process.

Beyond the fourth stage, the rice husks are processed according to the desired sample size. Although only 0.1 g to 0.4 g of activated carbon is required for the adsorption of 25 ml of 25 ppm Zn(II) ions and Cu(II) ions, each sample shall consist of 10 g of grinded rice husks. This is to cater for the weight loss anticipated after digestion and carbonization.

3.1.1 Washing

Washing is done to remove dust and other foreign body from the rice husks. It was observed that a large portion rice husks were still attached to tiny bits of rice grains while some of the rice husks were still attached to the paddy plant stem. Hence the washing of the rice husks also involved the removal of the bits of rice grains and the stems.

First the rice husks were placed in a 2 inch depth tray. While still dry, the rice husks were screened manually for the stems or sticks by running through them with fingers and picking out the stems and sticks. Once the obvious stems were removed, the rice husks were placed on a plastic sieve tray and rinsed with distilled water to get rid of the dust. The water that ran through the sieve was observed. Once the water looked clear, the rinsing was stopped and the rice husks were placed into the tray once again and soaked in distil water. At this stage, the unwanted rice grains were removed from the bottom of the tray. It was impossible to remove all the rice grains as they were quite fine and appeared in large quantities. Consequently, the rice grains were removed as much as reasonably possible. Lastly, the rice husks were rinsed.

3.1.2 Drying , Grinding and Sieving

The washed rice husks were placed in a 16-inch metal tray before being dried in the electric oven for 24 hours at 110°C .Once completely dried, the rice husks were milled using a laboratory blender. Since the blades of the blender randomly cut the rice husks, a mixture of different particle sizes were obtained. The intended particle size is 500 μm.

Hence the grinded rice husks were sieved through a set of sieve trays sized 2 mm, 1 mm, 0.5 mm, 250 microns, 125 microns and 100 microns on the sieve–shaker machine. From the sieving process, it was found that a larger portion of the grinded rice husks were of sizes 250 microns, 125 microns and 100 microns. Although the intended particle size was 0.5 mm, the amount produced during grinding was very little. Hence, to avoid wastage of raw material, a test was done to determine whether it was feasible to use rice husks with particle size of 250 microns, 125 microns and 100 microns instead of 0.5 mm. The test methodology will be discussed in Section. 4.1.

3.1.3 Digestion in Nitric Acid

In the digestion process, only sieved rice husks of particle size 0.5 mm were used. Each sample consisted of 10 grams of the rice husks, soaked in 3 M nitric acid. These samples were prepared in different nitric acid to rice husks weight ratios, namely 1:1, 1.5:1, 2:1 and 2.5:1. The following volumes of nitric acid were used to obtain the required weight ratios:

Table 4: Volume of Nitric Acid Required for Digestion of 10 g Rice Husks

Weight Ratio	Volume of Nitric Acid Required (ml)
1.0:1.0	52.9
1.5:1.0	79.36
2.0:1.0	105.8
2.5:1.0.	132.26

The rice husks samples were soaked for 20 hours in four separate 250 mL beakers sealed with parafilm to avoid any loss of nitric acid due to evaporation. After 20 hours each sample was rinsed with excess distil water until the pH of wash water was pH5. The pH was monitored by using a pH meter during the washing process. Once pH 5 was achieved, the samples were filtered with a filter paper and a funnel. The rice husks were then placed in Petri dishes which were later placed in the electrically heated oven at 110 °C for 4 hours.

3.1.4 Pre-Carbonization

While waiting for the rice husks to dry in the oven, four crucibles were weighed and labeled according to the different weight ratios. The furnace was set to 500 °C, 45 minutes prior to carbonization. This is because the temperature of the furnace rises very slowly and it takes at least 40 minutes for the temperature to reach 500 °C.

The rice husks samples were then collected from the oven and placed in the respective crucibles. The weight of the rice husks and crucibles (without lids) were measured and recorded to obtain the weight of dried digested rice husks.

3.1.5 Carbonization

The crucibles were covered with their lids and then placed into the muffler furnace one by one, with great care. The samples were left in there for 1 hour for samples A1, A2 A3 and A4, 2 hours for samples B1, B2, B3, B4, 3 hours for C1, C2, C3, C4 and 4 hours for D1, D2, D3 and D4. Number of samples carbonized each time is limited to four samples as the furnace is actually quite small.

The temperature of the furnace was lowered at least 15 minutes before the carbonization period is completed. This was to allow the furnace temperature to reduce, making it safer to remove the crucibles from the furnace. The resulting substance from the carbonization was then analyzed.

3.1.6 Analysis

The crucibles and their contents were left to cool before being weighed. The weight of activated carbon can be calculated by the following equation:

Weight of Activated Carbon	= *(Weight of Crucible+ Weight of Carbonized Rice Husks)* – *(Weight of Crucible)*	[Eq 3.1]

All carbonized samples were stored in air tight glass bottles according to their respective weight ratios and carbonization time. These glass bottles were then stored in a set of desiccators filled with silica gel to prevent any moisture from damaging the activated carbons.

3.1.7 Test to Determine Appropriate Particle Size of Rice Husks

The objectives of this test is to determine whether rice husks of sizes 250 microns, 125 microns and 100 microns can withstand the most extreme conditions of carbonization which will be used later in the experiment. It is also to select the most appropriate particle size.

2 g of rice husks from these sizes were digested in nitric acid with weight ratio of 2.5:1. The samples were left to digest for 20 hours before being rinsed in excess distilled water to maintain the pH at pH 5 and to remove the excess nitric acid. For the last rinse, the samples were filtered. The filter paper was left in the fume hood to dry overnight. Once the samples were dried, the weight was recorded and the samples were placed in 3 separate crucibles. The furnace was set at 700°C and the samples were placed inside the furnace. For the carbonization process, all three crucibles must be closed with lids to avoid open burning. The samples were left in the furnace for three hours. The temperature was then lowered and the samples were carefully taken out of the furnace.

The adsorption capacity for both metal ions were tested on a the effect of weight ratio of HNO_3 to rice husks, the effect of carbonization time, the contact time and also the size of sample loading. The next few paragraphs describe the steps in conducting the adsorption.

3.2.1 Effect of Weight Ratio and Carbonization Time

i. Pipette 25 ml of the 25 ppm $Zn(NO_3)_2$ solution into 16 separate 150 ml conical flasks.
ii. Weigh and insert 0.1g of activated carbons of each sample into the conical flasks. Label the conical flasks according to the respective sample names.
iii. Seal the conical flasks with parafilm to avoid splashes from occurring during adsorption process.
iv. Place each conical flask onto the shaker machine (GFL Model 3017) and set the speed of rotation as 150 rpm.
v. Secure the conical flasks by tightening the securing bar.
vi. Activate the shaker and leave the samples on the shaker for 2 hours.
vii. Filter the samples into separate polyethylene bottles.
viii. Analyze the filtrates of each sample by using the Atomic Absorption Spectrophotometer (Z-5000 Polarized Zeeman AAS, HITACHI) to obtain the residue concentration of Zn(II) ions in the filtrates.
ix. The amount of Zn(II) ions adsorbed per gram of activated carbon prepared is determined by taking the difference between the initial concentration of Zn(II) and the residual of Zn(II) ions in the filtrate.
x. The steps above were repeated by replacing $Zn(NO_3)_2$ with $Cu(NO_3)_2$ solution of the same volume and concentration. Please refer to Figure 3 for the experimental set up.

3.2.2 Effect of Contact Time

The procedures under section 3.3.1 were repeated for the activated carbon samples by shaking the samples for 1 hour, 3 hours and 4 hours. However, due to the time constraint, the adsorption was only done for the samples that adsorbed the most Zn(II) ion and Cu(II) ions in the previous section.

3.2.3 Effect of Activated Carbon Loading Size

i. The procedure in section 3.3.1 was repeated for the samples that adsorbed the highest amount of Zn (II) ions and Cu (II) ions during the first batch of adsorption.
ii. 0.2g, 0.3g and 0.4g of each sample were used instead of the 0.1g loading.
iii. The results of this experiment were compared with the result of the first batch of adsorption where by the loading size was 0.1g.

The experimental set up shown below was applied for the adsorption experiments mentioned in sections 3.2.1, 3.2.2 and 3.2.3.

Figure 3: Experimental set up for adsorption process

3.3 Tools and Equipment Used

Table 6 : Tools /Equipment Required for the Research Project

No	Tool/Equipment
1.	Electrical Oven (CARBOLITE)
2	Shaker machine (GFL Model 3017)
3.	pH meter
4.	Atomic Absorption Spectrophotometer (AAS)
5	Fourier Transform Infra Red Spectrophotometer (FTIR-8400S) SHIMADZU
6	Muffler Furnace (CARBOLITE)
7.	Electronic Weighing Scale (MONOBLOC)

3.3.1 Atomic Absorption Spectrophotometer (AAS)

The AAS is the main analytical equipment used in this research work as it is the only equipment that can give definite readings of the concentration of metal ions. Hence a good understanding of the basic functionality and built of the unit can be useful in the research.

Atomic absorption spectrophotometer works by providing the accurate quantitative analyses for metals in water, sediments, soils or rock. The samples are analyzed in the form of solution. Atomic absorption spectroscopy (AAS) is a process whereby electromagnetic radiation (EMR) is absorbed or emitted by atoms and measured. Since all atoms can absorb EMR, the wavelength at which EMR is absorbed or emitted is exclusive for a particular chemical element. Besides that, the AAS can detect many metal ions and a few non-metals in terms of quantity.

Atomics absorption units have four fundamental parts: interchangeable lamps that emit

light with element - specific wavelengths, sample aspirators, a flame (atom cell) or furnace apparatus for volatilizing the samples, and a photon detector. (http://departments.colgate.edu/geology/instruments/aa.htm)

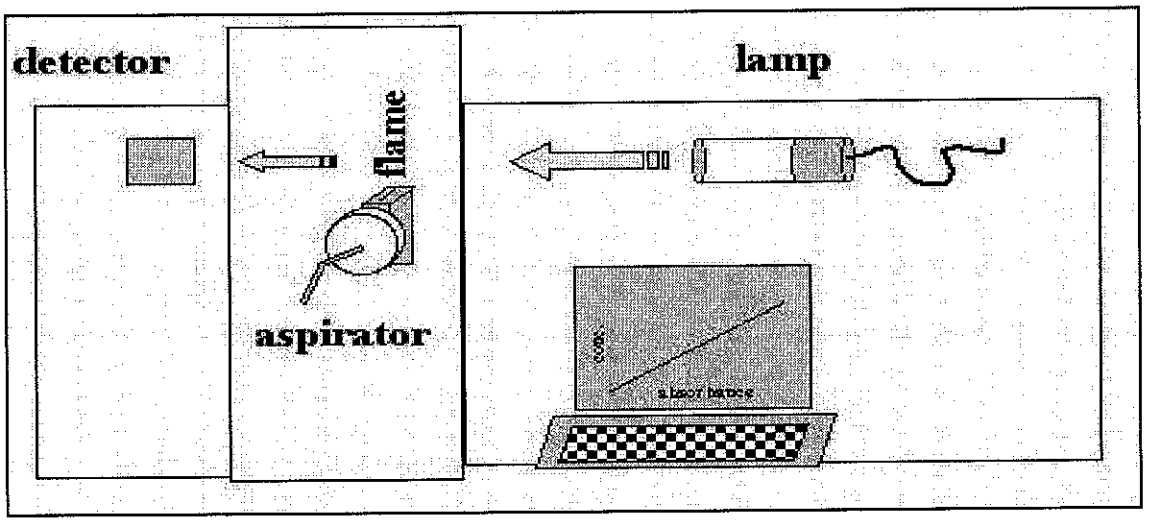

Figure 4: Schematic diagram of Atomic Absorption Spectrophotometer.

An atomic absorption spectrophotometer generates a source of light to be absorbed and passed through the gaseous state of the sample that has been sprayed into a flame and the analytes are determined by measuring the absorption of the constant intensity of EMR that passes through the flame as shown in Figure 4.

Typically the light source used is the hollow cathode lamp because that source of light can provide a narrow line source. A hollow cathode lamp is made of glass envelope with a cathode inside made of the element being analyzed and a suitable anode. Hollow cathode lamps are preferred because they are easy to use, they produce a stable and intense light, they are economical and that hollow cathode lamps can be made for all chemical elements detected by the atomic absorption spectrophotometer.

The atom cell commonly used is the flame that is distinguished by the gases involved, the temperature of the flame, the flow of gases, the form in which the gases are mixed, its shape and size. Oxy- acetylene at temperature 2003°C and nitrous oxide-acetylene at 3000°C are the most typically used flames. Atoms are formed within the flame by the

atomization process. The dissolved sample in solution is heated to sufficiently high temperatures to decompose into compounds, that eventually further decomposes into individual atoms. The atoms in the gaseous state will then absorb the EMR from the light source.

To detect the atoms, a photomultiplier tube emits electrical signals which are proportional to the intensity of EMR of the wavelength separated by the light dispersion monochromater. These signals will then be transferred to the computer and the output will be seen on the monitor of the computer in terms of concentration (ppm).

3.3.2 Fourier Transform Infrared (FTIR) Spectroscopy

The Infrared (IR) spectroscopy is widely used to determine the structure of organic compounds. IR can identify the presence of certain functional groups within a molecule. The FTIR technique is an analytical method used to identify the chemical structure of many inorganic chemicals and also organic materials or compounds either quantitatively or qualitatively. This equipment records down the infrared absorbency of a sample. The frequencies at which the absorption occurs will indicate the type of the functional groups present in the sample.

To run the analysis, a small amount of powdered sample was mixed with KBr (MERCK) before being compressed into a disc. The disc was inserted onto the sample compartment and the spectra of the sample were recorded. Most FTIR units operate within the range of 4000-400 cm^{-1} wave number. The wave number is used as these numbers are directly proportional to energy (Atkins et. al, 2002). A higher wave number light has more energy than that of a low wave number light. For example, 3000 cm^{-1} light has more energy than 500 cm^{-1}. Functional groups (the chemical structure fragments within molecules) tend to absorb a photon of infrared radiation. Hence the energy increase and is associated with bond vibrations involving in stretching and bending of bonds. In this experiment, the FTIR was used to detect the vibration

frequency changes in the activated carbon. The spectra for one of the samples shall be examined in Chapter 4,

3.4 Chemicals and Materials Used

Table 7: List of Chemicals and Materials

No.	Chemicals/Material
1.	Nitric acid (65% by SYSTERM)
2.	Rice husks
3.	Zinc(II) Nitrate (SYSTERM)
4.	Copper (II) Nitrate (SYSTERM)
5	Deionized Water
6	Zn (II) ions standard solution, 1000 ppm (FISCHER)
7	Cu (II) ions standard solution, 1000 ppm (FISCHER)

3.4.1 Standard Solutions

The standard solutions of 1000 ppm for both Copper(II) and Zinc(II) ions are diluted to specific concentrations to form the calibration curve for the AAS. The table below shows the concentration of standard solutions required for the AAS unit.

Table 8: Usage of Standard Solutions

Brand of AAS Unit	**HITACHI**	**SHIMADZU**
Concentrations of Copper Standard Solution (ppm)	1, 2, 4	0, 1, 10, 25
Concentration of Zinc Standard Solution (ppm)	0.2, 0.4, 0.8	0, 1, 10, 25
Choice of Concentration	Fixed	Depends on the expected range of concentration

CHAPTER 4

RESULTS AND DISCUSSION

This chapter will present the results and discussion for three main aspects of the research work, namely the preparation of activated carbons, the adsorption capacity of the activated carbon towards Zn (II) ions and Cu (II) ions at different conditions.

4.1 Production of Activated Carbon

4.1.1 Appropriate particle size selection

In the early stage of the research, the author needed to determine whether rice husks of particle sizes 250μm, 125μm and 100μm could withstand the extreme conditions of the activated carbon process. The following parameters were used:

Table 9 : Parameters for particle size test

Temperature ^{o}C	Carbonization Duration (hr)	Particle size (μm)
700	3 hours	500
		250
		125
		100

All three samples except for that of 500 μm were burnt into ashes. The three samples appeared as white powdery substances. This meant that activated carbon was not produced at all. From this result, it was concluded that the rice husks with sizes of 250 μm, 125 μm and 100 μm cannot withstand the high temperature of 700°C and the duration of carbonization of 3 hours. Theoretically rice husks ashes have no cellulosic materials and produced silica with a slightly lower specific surface area (Rahman, et.al , 2000). However, it is not in the scope of this study to evaluate the adsorption capacity of rice husks ash. Therefore, particles of these three sizes will not be used and particles size of 0.5mm will be used. The picture in Figure 5 below shows the ash that was formed for particle sized 100 μm.

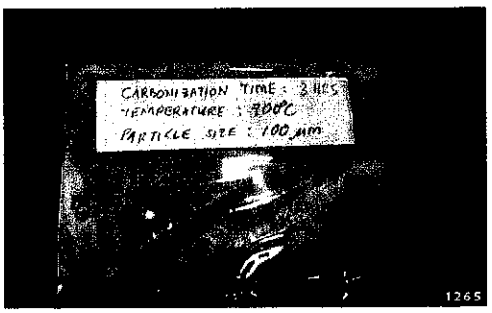

Figure 5: Ash produced from 100 μm rice husk particles

4.1.2 Digestion with Nitric Acid

The digestion was done in nitric acid, 3 M for 20 hours, using acid to rice husks weight ratios of 1.0:1.0, 1.5:1.0, 2.0:1.0, and 2.5:1.0. To obtain these weight ratios, the following volumes of nitric acid, 3 M are added to 10 g of rice husks per sample according to Table 4, page 14.

After digestion, the rice husks appeared to be yellowish dark brown in color. Most of the particles would sink down to the bottom of the beaker containing the nitric acid and rice husks. The nitric acid in which the rice husks were soaked in also was yellowish in color. The nitric acid was also slightly cloudy and some foam could be seen on the surface along with a small amount of rice husks floating on the surface. Samples for all four weight ratios showed the same physical appearance after digestion. The picture below shows the condition of the digested rice husks.

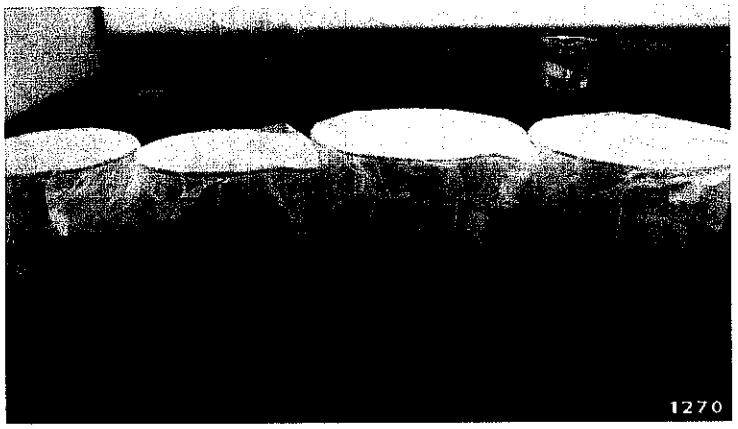

Figure 6: Digested Rice husks after 20 hours

In terms of weight, the rice husks had shown about weight loss of 4.8% to 11.8%. On average, the percentage weight loss was 8.68 %. These percentages were calculated from the equations below and the results are tabulated in Table 10.

$$\text{Weight of Digested Rice Husks} = (\text{Weight of Crucible + Digested Rice Husks}) - (\text{Weight of Crucibles}) \quad \text{[Eq. 4.1]}$$

$$\text{Percentage Weight Loss} = \frac{(\text{Weight of Rice Husks}) - (\text{Weight of Digested Rice Husks})}{(\text{Weight of Rice Husks})} \times 100\% \quad \text{[Eq. 4.2]}$$

Table 10: Results of Rice Husks Digestion

Sample Name	HNO_3 to Rice Husks Weight Ratio	Weight of Rice Husks Initially (g)	Weight of Digested Rice husks (g)	Percentage Weight Loss After HNO_3 Digestion (%)
A1	1.0:1.0	10	9.05	9.5
A2	1.5:1.0	10	9.09	9.1
A3	2.0:1.0	10	9.01	9.9
A4	2.5:1.0	10	8.96	10.4
B1	1.0:1.0	10	9.21	7.9
B2	1.5:1.0	10	9.07	9.3
B3	2.0:1.0	10	9.23	7.7
B4	2.5:1.0	10	9.10	9.0
C1	1.0:1.0	10	9.51	4.9
C2	1.5:1.0	10	9.52	4.8
C3	2.0:1.0	10	9.37	6.3
C4	2.5:1.0	10	9.41	5.9
D1	1.0:1.0	10	8.96	10.4
D2	1.5:1.0	10	8.94	10.6
D3	2.0:1.0	10	8.86	11.4
D4	2.5:1.0	10	8.82	11.8

The weight loss was anticipated as previous studies have shown significant weight loss of rice husks after digestion. This is due to the loss of hemicellulose, cellulose and lignin that were "readily digested by nitric acid." The digested constituents were dissolved in the nitric acid. The remaining weight is made up of silica which is almost insoluble in nitric acid (Rahman et.al, 2000).

It was also observed that the digestion reaction was a vigorous and exothermic reaction. Some heat could be felt on the surface of the beaker during the digestion periods. According to previous works by Rahman, Ismail and Osman, the typical percentage weight loss was 68% to 74%. This differs from the results obtained by Ng, (2004) where by the percentage weight loss was about 20 % (Ng, 2004).

In contrast, the author's results of post-digestion weight loss differed completely from the previous works in terms of the magnitude of weight loss. This may be due to the fact that the digestion was done in sealed beakers rather than in open air. This meant that there is a possibility that in previous works, the digestion was done in open containers and some of the dissolved components had evaporated into the air. On the other hand, the samples digested by the author were sealed up so that no evaporated molecules could escape to the air. As a result, it can be speculated that these dissolved components actually settled back into the rice husks. This can explain the reduction of percentage of weight loss compared to previous works. Figure 7 below shows the graph of percentage weight loss against the weight ratio of nitric acid to rice husks.

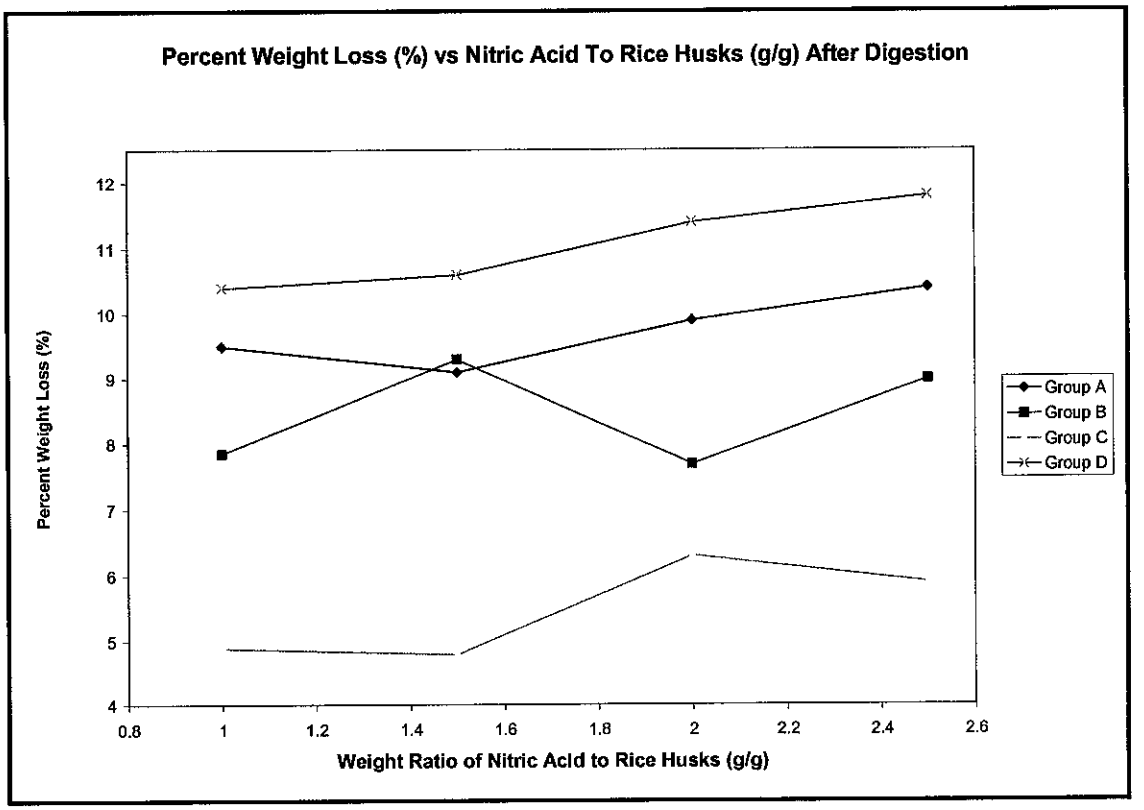

Figure 7: Graph of Weight Loss (%) Vs. Weight Ratio of Nitric Acid to Rice Husks (g/g) After Digestion with Nitric Acid 3M

From the graph above, Group A, Group B, Group C and Group D represent the samples that would be carbonized for 1 hour, 2 hours, 3 hours and 4 hours respectively. However, at this point, all samples had yet to be carbonized. Hence, the time of

carbonization would not affect the results of the digestion process. Based on the graph, it may be concluded that an increment of weight ratio may increase the percentage of weight loss. This is apparent for samples under Group D as the percentage of weight loss increased steadily with the increment of the weight ratio. Although samples under Group A, B and C did not show a steady increase in percentage weight loss, each of these samples showed a net increase of percentage weight loss. The trend exhibited by the samples in Group D was similar to the trend produced in the research of Ng. Joon Leong (2004). It was expected that the samples of Groups A, B and C should also follow the same trend. However, this was not the case. The discrepancy in pattern was probably due to the loss of some amount of rice husks during the water rinsing of the digested rice husks to achieve pH5.

Based on the research by Ng, (2004), the percentage weight loss of rice husks should increase with the increment of nitric acid to rice husks weight ratio. Nitric acid causes weight loss in rice husks as it readily dissolves organic compounds such as cellulose, hemicellulose and lignin. At higher weight ratios, the amount of nitric acid molecules per gram rice husks is also higher. Thus more nitric acid molecules will react with that same amount or rice husks compared to that of a lower weight ratio. The more nitric acid molecules act on a sample, the more organic compounds are dissolved in the acid, increasing weight loss. This explains the higher weight loss percentage for samples with weight ratio of 2.5:1 compared to that of 1:1.

4.1.3 Carbonization

Initial Efforts

Initially this process, also known as pyrolysis was done at 500 °C on samples weighed 1 g each. The appearance of the activated carbons can be seen in Figure 8, Figure 9 and Figure 10. Twelve samples, each weighing 1 g were produced. Samples A1, A2, A3 and A4 were carbonized for 1 hour, B1, B2, B3 and B4 for 2 hours and C1, C2, C3 and C4 for 3 hours. The results of the initial carbonization process are shown in Table A.II.1 under Appendix II.

The pyrolysis must be done in absence of oxygen to avoid the formation of ashes in the activated carbon. Since the muffler furnace available did not have the feature of providing inert gas flow, the lids of the crucibles were closed as tightly as possible to avoid ingress of oxygen into the crucibles. Although the lids were closed during carbonization, there was still a little ash produced. This may be due to the uneven surface of the crucibles' lids or the rim of the crucibles that allowed air to enter. These samples were not used for the adsorption process due to the fact that the initial weights of the samples reduced dramatically after carbonization. Hence, the amount or activated carbon left behind was insufficient for the adsorption experiments.

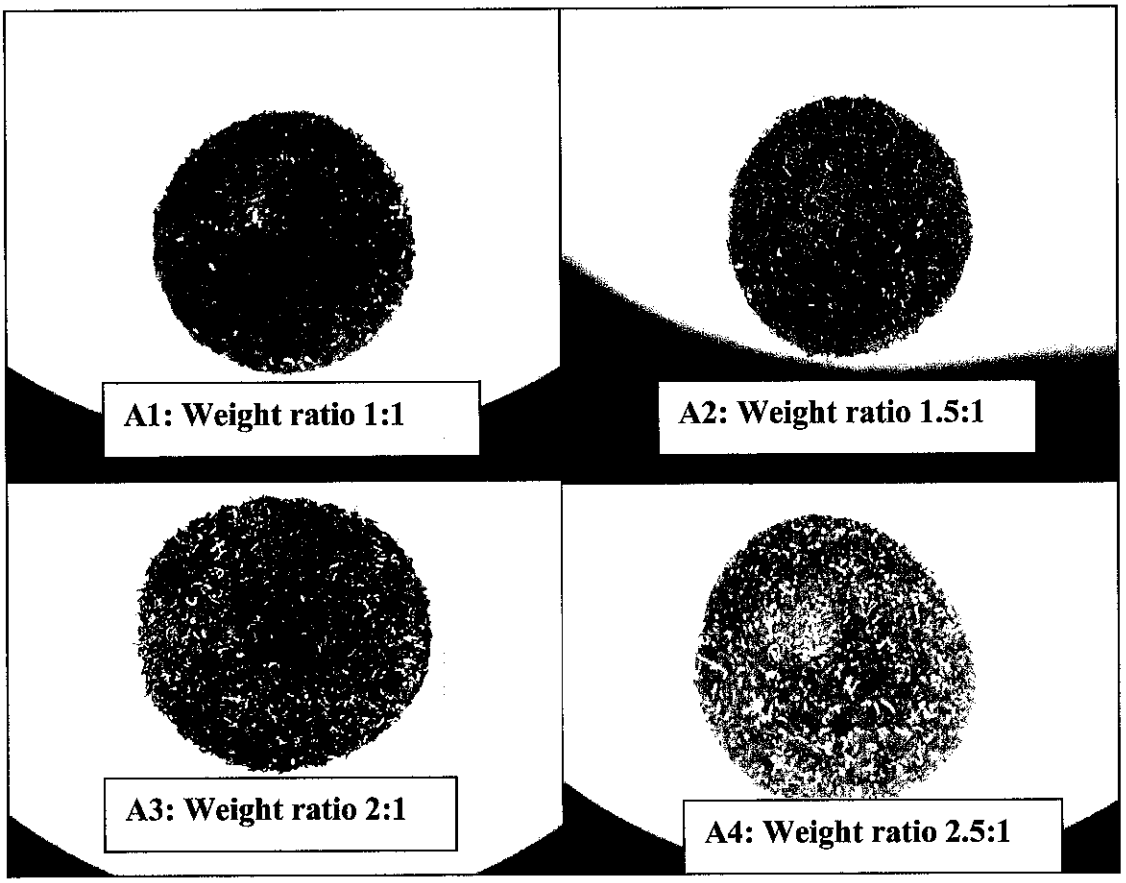

Figure 8: Rice Husks Carbonized at 500 ^{0}C for 1 hour

Figure 8 shows Samples A1, A2, A3 and A4 for 1 hour with the weight ratios acid to rice husks 1: 1, 1.5:1, 2:1 and 2.5:1, respectively. The white substances in the samples are the ashes formed due to the presence of oxygen during the carbonization process.

For weight ratios 1:1 and 1.5:1, the ash content was almost negligible. However with increasing weight ratios, the ash content appears to increase as well. On the whole, the ash content is the highest for weight ratio 2.5:1 followed by weight ratio 2:1. Nevertheless, the carbonization process for this group was successful as the ash content was relatively low. Unfortunately, the activated carbon yield was low, ranging from 16.2 % to 32 % and the highest yield is that of weight ratio 2:1.

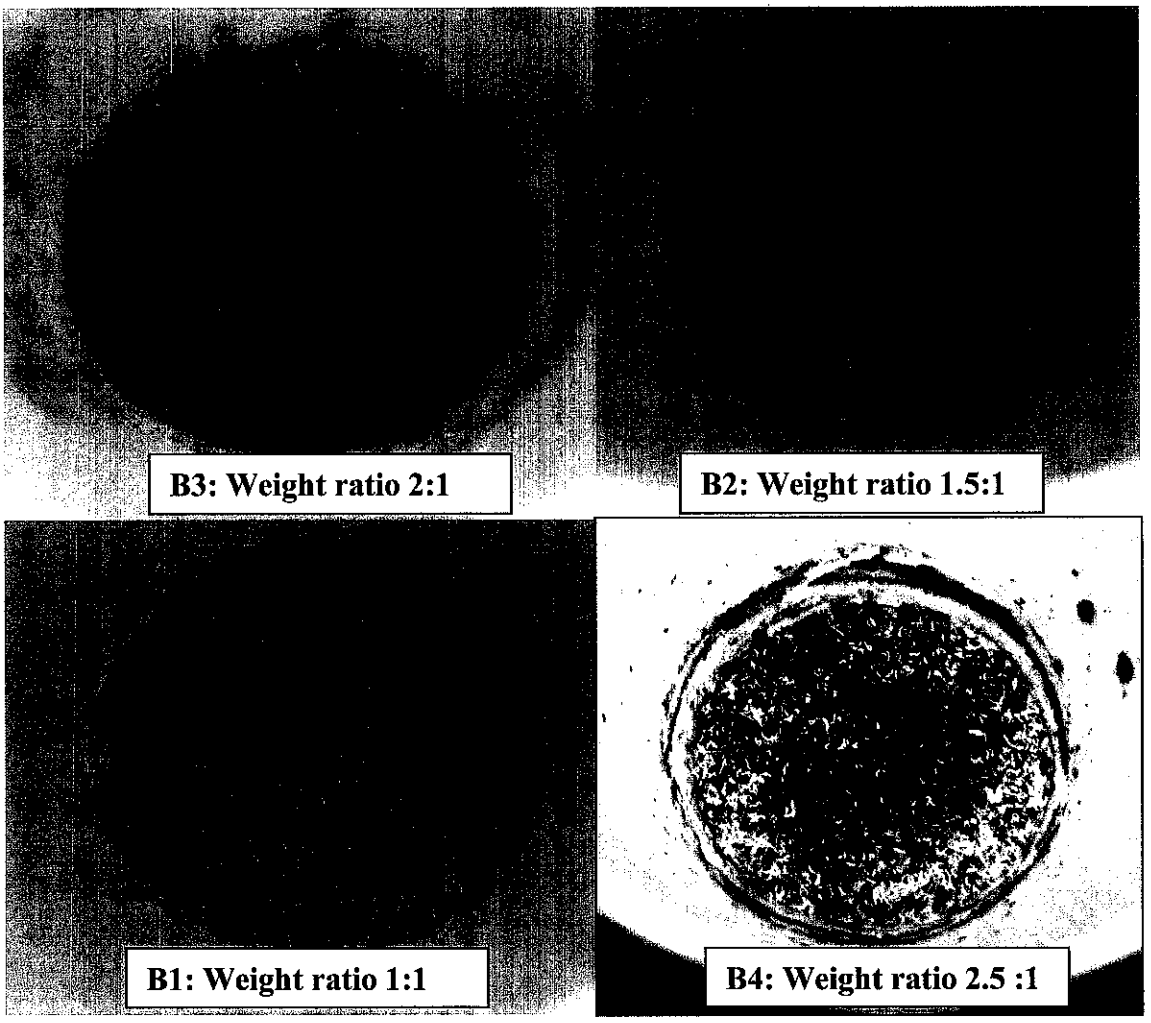

Figure 9: Rice Husks Carbonized at 500°C for 2 hours

Figure 9 shows the rice husks carbonized for two hours at 500°C. The activated carbon produced increased in ash content compared to samples A1, A3 and A4 that were carbonized for 1 hour. The samples with least ash content was still of weight ratio 1:1 followed by 1.5:1. However, the sample with the most ash content is B3, with weight

ratio 2:1. This differs from the trend shown in set A where samples were carbonized for 1 hour and the highest ash content was that of weight ratio 2.5:1.

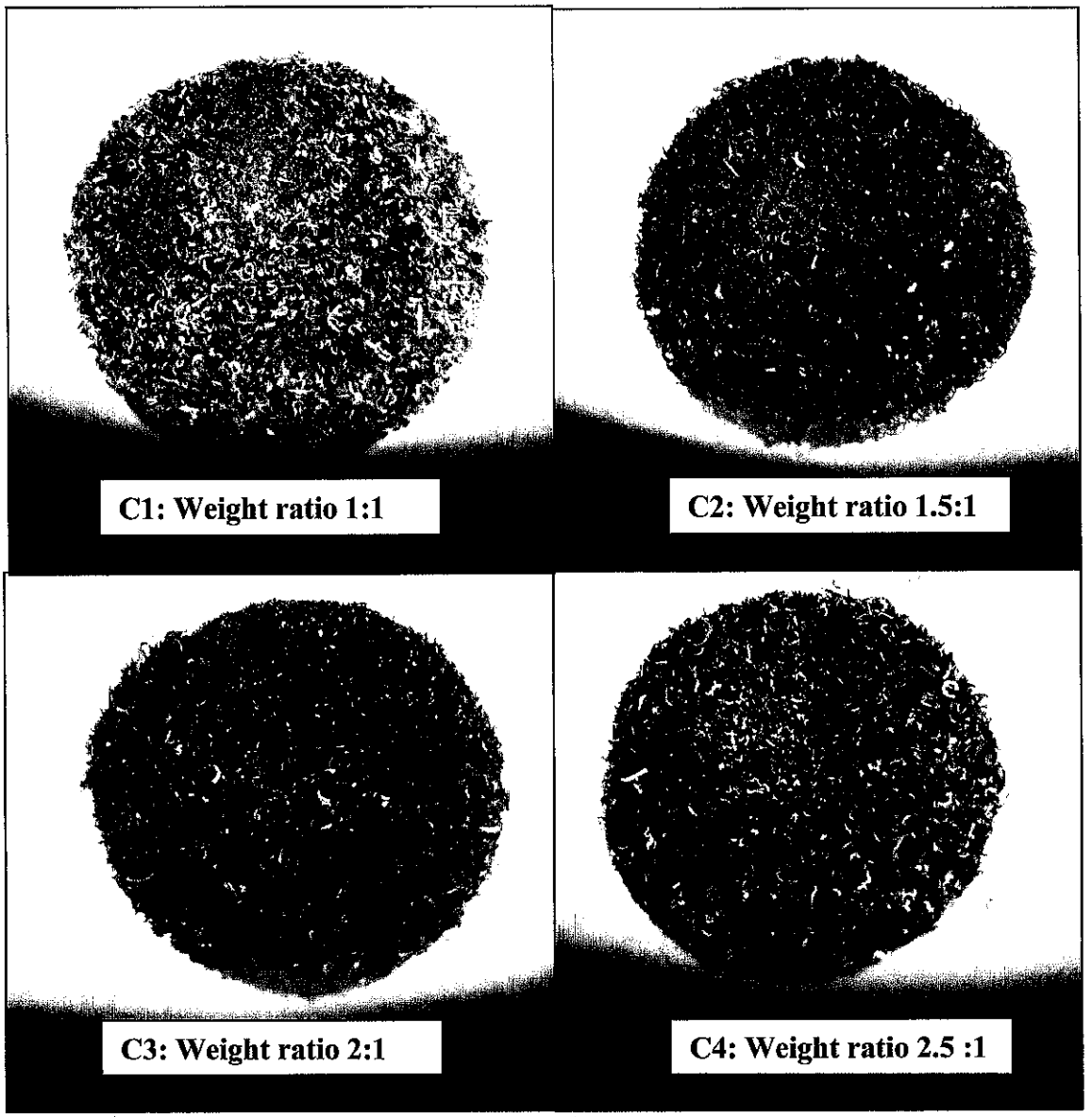

Figure 10: Rice Husks Carbonized at 500°C for 3 hours

Figure 10 shows the rice husks after 3 hours of carbonization. Sample C1, C2, C3 and C4 have weight ratios of 1:1, 1.5:1, 2:1, and 2.5:1 respectively. In terms of ash content, C2 has the least ash followed by C3. The most ash content is that of sample C1. The trend of ash content in this set differs from the Set A and Set B as the ash content of

each sample in Set C does not differ much from each other. Unlike the Set A and Set B, the sample with weight ratio 1:1 has the most ash content.

In terms of weight, sample C2 has the highest yield of 4.95%. The yields of the other three samples are the same, 3.96%. The yield for all four samples is very low. Therefore, for future activated carbon preparation, at least 10 g of rice husk shall be used in order to produce at least 0.1 g activated carbon.

Final Attempt in Producing Activated Carbons

For the second batch, 16 samples were produced and labeled as tabulated in the table below.

Table 11: Activated Carbon Produced At Different Parameters

		Weight Ratio of Nitric Acid to Rice Husks (g/g)			
		1.0	1.5	2.0	2.5
Carbonization Time (hr)	1	A1	A2	A3	A4
	2	B1	B2	B3	B4
	3	C1	C2	C3	C4
	4	D1	D2	D3	D4

Each sample was produced out of 10 g of rice husks at particle size of 0.5 mm. The activated carbons produced for all samples were free from ash. This may be due to the reason that the crucibles used were the same size as those used in the previous batch. Compared to the previous batch, the volume that was filled up by each sample in the new batch was 10 times larger than the volume occupied by the previous batch. This meant that, should there be any presence of oxygen ingresses through lids of the crucibles; the volume of oxygen that could fill up the remaining area above the rice husks surface was lower compared to that of the previous batch. As all the samples in

free and appeared the same physically, only one of the samples in this batch was photographed as shown in the figure below.

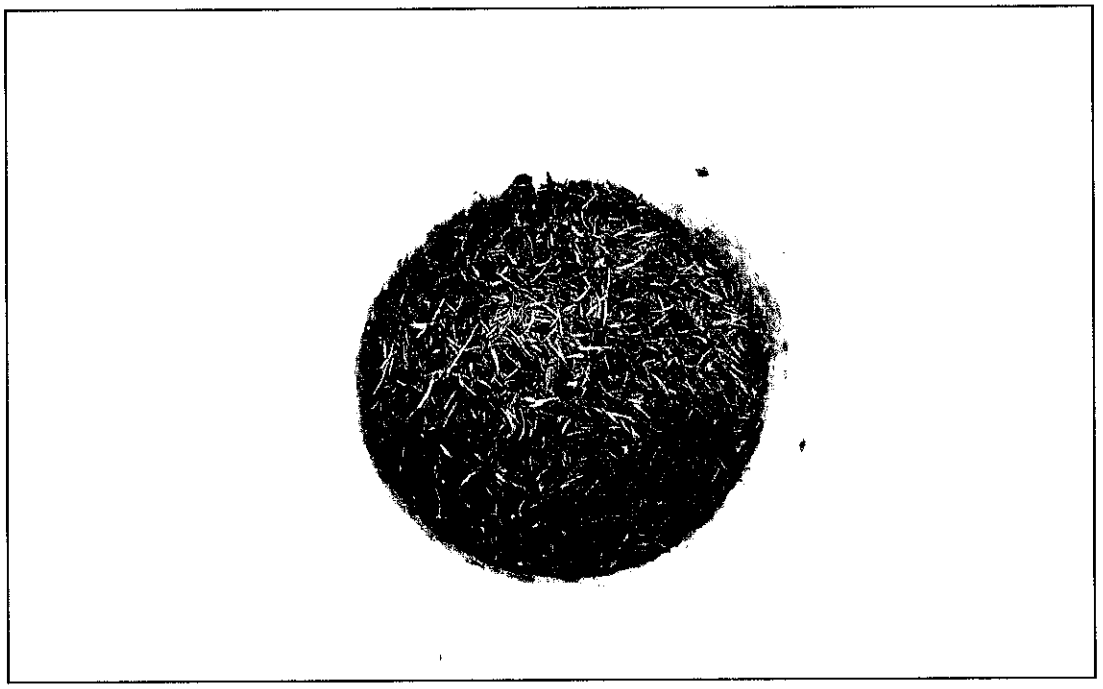

Figure 11: Rice Husks Successfully Carbonized at 500°C

The results for the successful batch of activated carbon are summarized in Table 12 shown in the next page. From the table it can be concluded that pyrolysis or carbonization actually reduces the initial weight of the rice husks tremendously. In this experiment, the yield of activated carbon was ranging from 28% to 37.77%. This was not alarming as previous research by Williams discovered that pyrolysis of rice husks at 500°C could yield 37 % oil, 17% water, 17.5 % gas and 29 % char, in terms of weight percent (Williams et. al, 2000).

Since the range of activated carbon yield did not deviate too much from 29 % of char, it can be concluded that the activated carbon yield in this experiment was well within the expected range.

Table 12: Result of Activated Carbon Yield

Sample	Initial Weight of Rice Husks (g)	Weight of Activated Carbon Produced (g)	Activated Carbon Yield (%)
A1	10	3.46	34.60
A2	10	3.37	33.70
A3	10	3.30	33.00
A4	10	3.21	32.10
B1	10	3.78	37.77
B2	10	3.26	32.57
B3	10	3.15	31.48
B4	10	3.28	32.78
C1	10	3.05	30.50
C2	10	2.96	29.60
C3	10	3.17	31.70
C4	10	2.9	29.00
D1	10	2.8	28.00
D2	10	2.928	29.28
D3	10	2.94	29.4
D4	10	2.87	28.7

4.2 Characterization of Activated Carbons Produced by Using FTIR Analysis

Basically, a molecule can absorb radiation through three processes, namely rotation, vibration and stretching. The spectrum's intensity is directly proportional to the atomic masses and inversely proportional to chemical bonds. The positions of peaks are used to identify atoms in a molecule, molecular structure and bonding between the atoms. Stretching vibrations of many functional groups such as C-H, O-H, and C=O and -NH are commonly found in the region of 4000 to 1600 cm^{-1}. The region from 1400 to 625 cm^{-1} is called the fingerprint region as they are very distinct for each material. No two compounds have the exact pattern of peaks (Atkins et. al., 2002). The following figures

show the FTIR spectrum for sample D4. For a clearer view of the spectra, please refer to Appendix III.

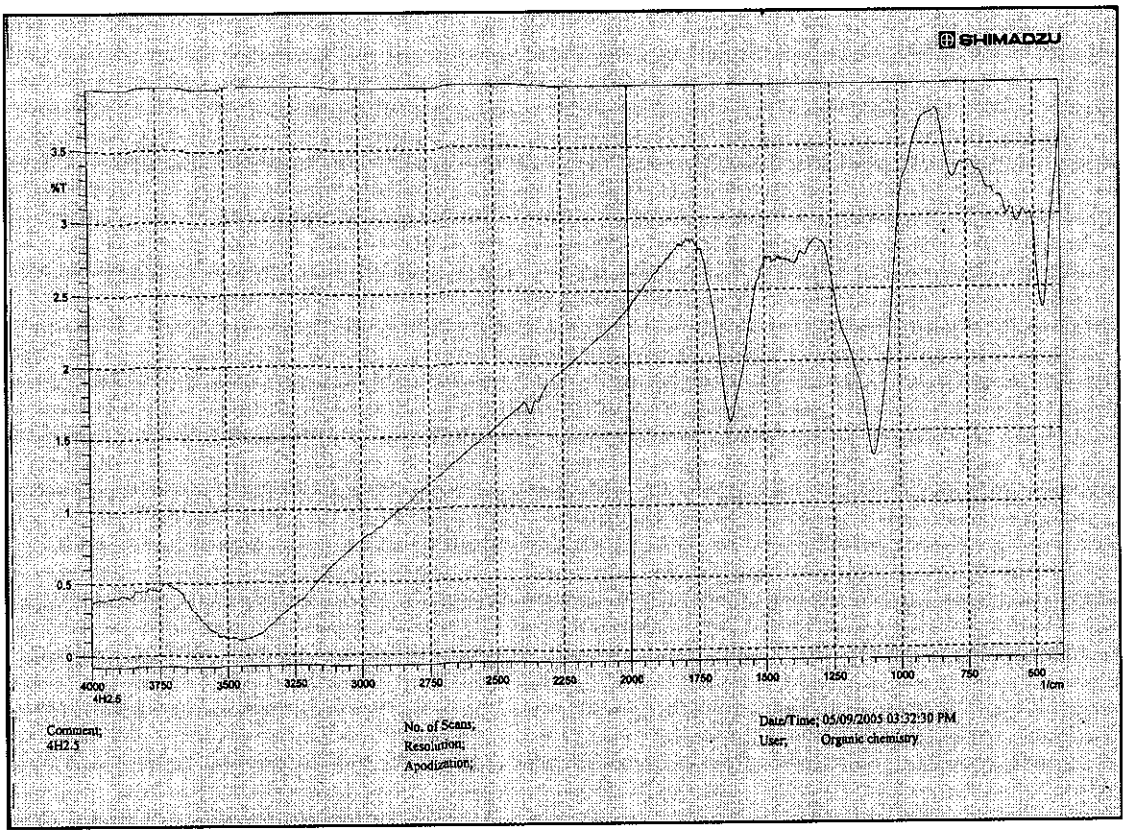

Figure 12: FTIR Spectra for Activated Carbons Made From Rice Husks

From the FTIR spectra, there is a broad band between 3000 cm^{-1} to 3755.15 cm^{-1}. This encompasses the region that represents the bonded hydroxyl (-OH) and amino (-NH) groups. Between 3000 cm^{-1} to 3500 cm^{-1} of the spectrum, the activated carbon exhibits a band corresponding to the stretching of -OH groups. This band is broad due to the hydroxyl bonds. The -OH band occurs at the same band as the stretching NH band.

The band identified as 2931.60 cm^{-1} shows the presence of -CH stretch. Ascending the curve, the next band was 2368.42 which falls within the range of 2500 cm^{-1} to 3600 cm^{1}. This range indicates carboxylic acid (-OH). This was followed by 1629.74 cm^{-1} which indicate the presence of carboxylate ions. The band at 1386.72 cm^{-1} is a weak stretching band caused by the carboxylate ions, followed by 1099.35 cm^{-1} which is due

to the -CO stretching of alcohol groups and also due to C-N vibrations (Ping et. al., 2003). Lastly, the band 798.47 cm^{-1} represents alkene.

Based on these identifications, the activated carbons from rice husks contain functional groups such as carbonyl, carboxyl, hydroxyl and amino. These functional groups can bind the heavy metals. The amino, a type of protein found in biomass is actually carboxylic acids that contain either acidic or basic side chain of amine functional group (Atkins et. al., 2002).

Amines derived from plants are called alkaloids. All amines can act as proton acceptors in hydrogen bonding to water molecules and they are the strongest bases of all neutral molecules (Atkins et. al., 2002).

Another functional group is the carboxyl group. This group acts as negatively charged carboxylate ligands which attract the positively charged metal ions such and Zn (II) and Cu(II) and binding occurs. (Baig et. al., 1999). Similarly, other groups such as carbonyl and alcohol may be also involved in metal binding. Therefore, these functional groups could be the agents of metal adsorption.

4.3 Adsorption Capacity Studies of the Prepared Activated Carbon

This section will be divided into two parts, namely the adsorption capacity of activated carbon towards Zn (II) ions and the capacity of the same samples on Cu (II) ions.

4.3.1. Effect of Carbonization Time on Zn (II) Adsorption

The table below summarizes the ultimate results that were obtained from the Atomic Absorption Spectrophotometer (AAS) for adsorption process of 2 hours duration. The detailed results and calculations leading to the values of adsorption capacities are detailed in Appendix IV.

Table 13: Adsorption capacity of Zn (II) ions at varied carbonization time and weight ratio.

Sample Name	Carbonization Time(hr)	Acid To Rice Husks Weight Ratio	Amount of Zn (II) adsorbed (mg)	Adsorption capacity (mg Zn (II) ions adsorbed/g carbon)	Percent of Zn(II) ions adsorbed (%)
A1	1	1.0	0.5633	5.6213	91.21
A2	1	1.5	0.5635	5.6070	91.26
A3	1	2.0	0.5633	5.6157	91.21
A4	1	2.5	0.5633	5.6325	91.21
B1	2	1.0	0.5643	5.6369	91.38
B2	2	1.5	0.5613	5.5901	90.89
B3	2	2.0	0.5608	5.5741	90.81
B4	2	2.5	0.5623	5.5945	91.05
C1	3	1.0	0.5585	5.5794	90.45
C2	3	1.5	0.5593	5.5925	90.57
C3	3	2.0	0.5593	5.5591	90.57
C4	3	2.5	0.5578	5.5608	90.32
D1	4	1.0	0.5580	5.5578	90.36
D2	4	1.5	0.5585	5.5794	90.45
D3	4	2.0	0.5575	5.5473	90.28
D4	4	2.5	0.5583	5.5825	90.40

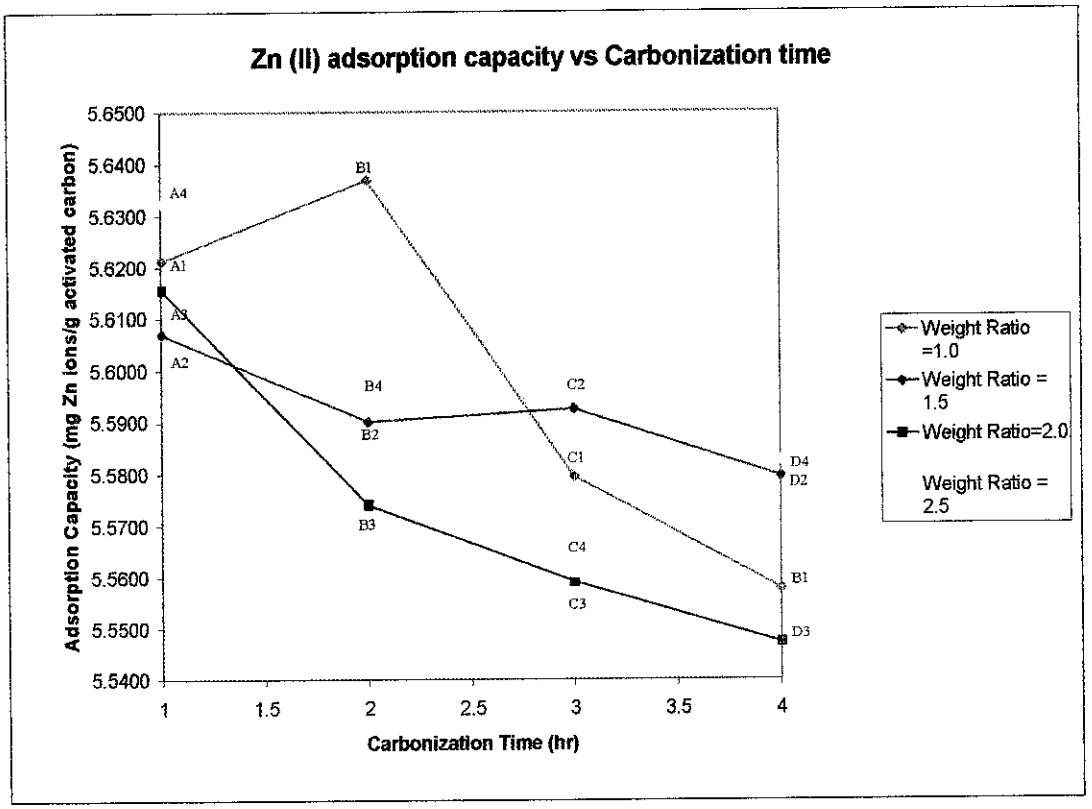

Figure 13: Adsorption Capacity vs. Carbonization Time

From the figure above the results indicate that beyond one hour of carbonization, there is no improvement on adsorption capacity. This is true except for samples with weight ratio 1:1 and carbonization time of 2 hours, B1. This sample yielded the highest amount of zinc ions among all 16 samples. Generally, the obvious trend is that for carbonization time above 1 hour, the adsorption capacity is reduced. However, for sample D4, the adsorption capacity seemed to have improved a little with the increment of carbonization time to 4 hours.

This result is in contrary with the conclusion drawn by Ng, (2004) regarding the relationship between carbonization time and adsorption capacity. According to his study, under specific weight ratio of nitric acid adsorption capacity is increased when the carbonization time is increased.

On the other hand, in the work of Castro et. al, (2002), using sugar cane bagasse and also residue sugar cane as raw material for preparing activated carbon, the effect of

carbonization time on the developed surface areas and pore volumes were studied. It was then observed that for both raw materials used, the S_{BET} (m^2/g) increases with carbonization time to a maximum value before decreasing with longer carbonization time. The same is said for the total pore volume and micro porous volume. In addition, the relative proportion of mesopores increased with increasing carbonization time.

According to Castro et.al, (2002) at short carbonization time, the carbon structure has not achieved a full development of porosity. Hence, a highly micro porous activated carbon was obtained. As the carbonization time increases, greater development of porosity was attained. By referring to the results published by Castro et. al., (2002), the mesoporous volumes increase with the carbonization time to a maximum value before decreasing with increasing carbonization time. In the same report by Castro et.al., it was reported that prolongation of carbonization time beyond 1 hour can lead to a pronounced reduction in porosity development, as reflected by the decrease in surface areas and total pore volumes of activated carbons from precursors. This is due to the break down of cross-link between carbon structures. This means that beyond 1 hour of carbonization, the adsorption capacity is decreased. This explains why the results obtained by the author showed decreasing adsorption performance as carbonization time was increased. Indeed the range of carbonization time experimented by the author was between 1 hour to 4 hours. This is outside the range of which the carbonization time could help the adsorption performance.

From this experiment, the author is now aware that although increasing time of carbonization can help increase adsorption capacity; there is also a maximum time limit of which any period beyond that would yield an opposite effect.

4.3.2. Effect of Carbonization Time on Cu (II) Adsorption

Table 14: Adsorption capacity of Cu (II) ions at varied carbonization time and weight ratio

Sample Name	Carbonization time (hr)	Weight ratio of HNO_3 to rice husks (g/g)	Amount of Cu (II) adsorbed (mg)	Adsorption capacity (mg Cu (II) ions adsorbed/g carbon)	Percent of Cu(II) ions adsorbed (%)
A1	1	1.0	0.3780	3.7762	60.48
A2	1	1.5	0.3723	3.7225	59.56
A3	1	2.0	0.3720	3.7052	59.52
A4	1	2.5	0.3583	3.5825	57.32
B1	2	1.0	0.2993	2.9865	47.88
B2	2	1.5	0.3265	3.2650	52.24
B3	2	2.0	0.3168	3.1612	50.68
B4	2	2.5	0.3285	3.2719	52.56
C1	3	1.0	0.4050	4.0299	64.80
C2	3	1.5	0.4070	4.0619	65.12
C3	3	2.0	0.3798	3.7861	60.76
C4	3	2.5	0.4000	3.9841	64.00
D1	4	1.0	0.4543	4.5380	72.68
D2	4	1.5	0.4215	4.2066	67.44
D3	4	2.0	0.4523	4.5135	72.36
D4	4	2.5	0.4598	4.5837	73.56

The effect of carbonization time on copper (II) adsorption differed from that of zinc (II) adsorption. For copper, there is a distinct pattern of which its adsorption capacity is the lowest at 2 hours carbonization time and continued to increase until its maximum capacity at 4 hours carbonization time. This trend was followed by all four different weight ratios as shown on Figure 14.

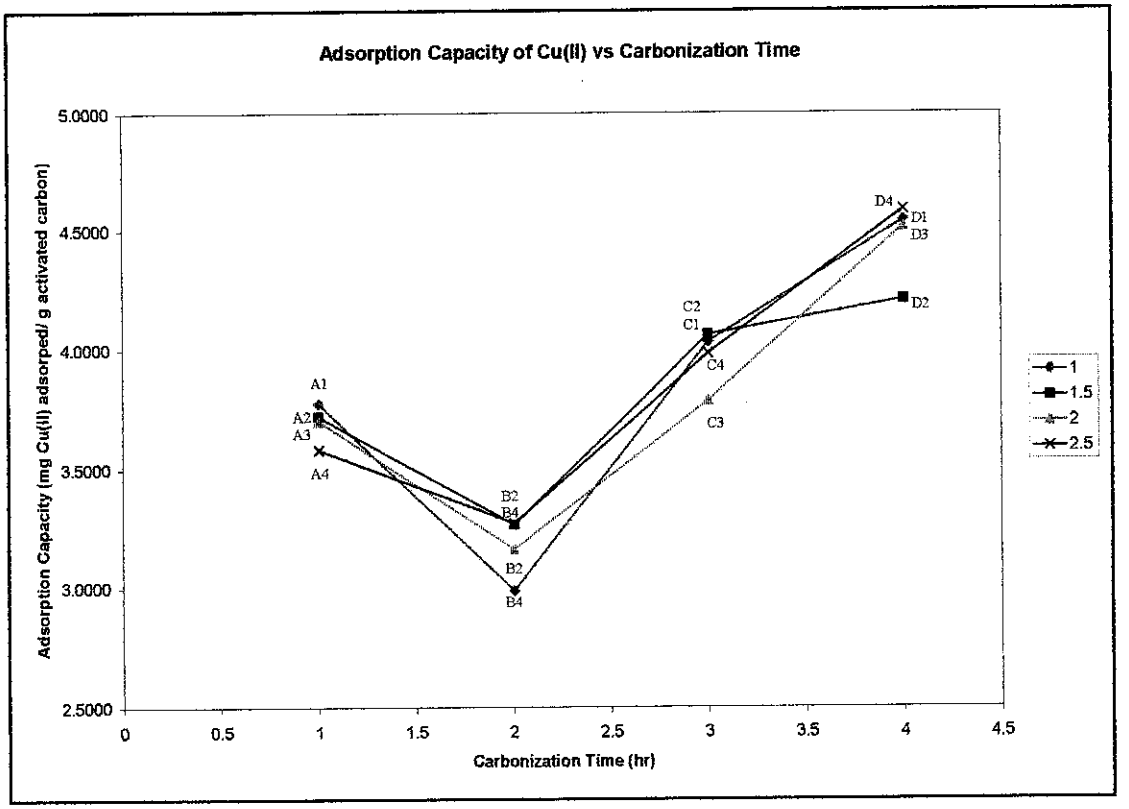

Figure 14: Adsorption Capacity of Cu (II) vs Carbonization time

4.3.3 Effect of Nitric Acid to Rice Husks Weight Ratio on Zn (II) Adsorption

Prior to the AAS readings, it was first speculated that the higher the weight ratio of acid to rice husks, the better the adsorption capacity. However, this was not the case.

The effect of weight ratio of acid to rice husk on the adsorption capacity of Zn (II) ions is not very apparent at higher carbonization times. It is however apparent at lower carbonization time. Referring to the following graph in Figure 15, increasing the weight ratio does not necessary increase the adsorption capacity of Zn (II) ions, this may be explained in the following paragraphs.

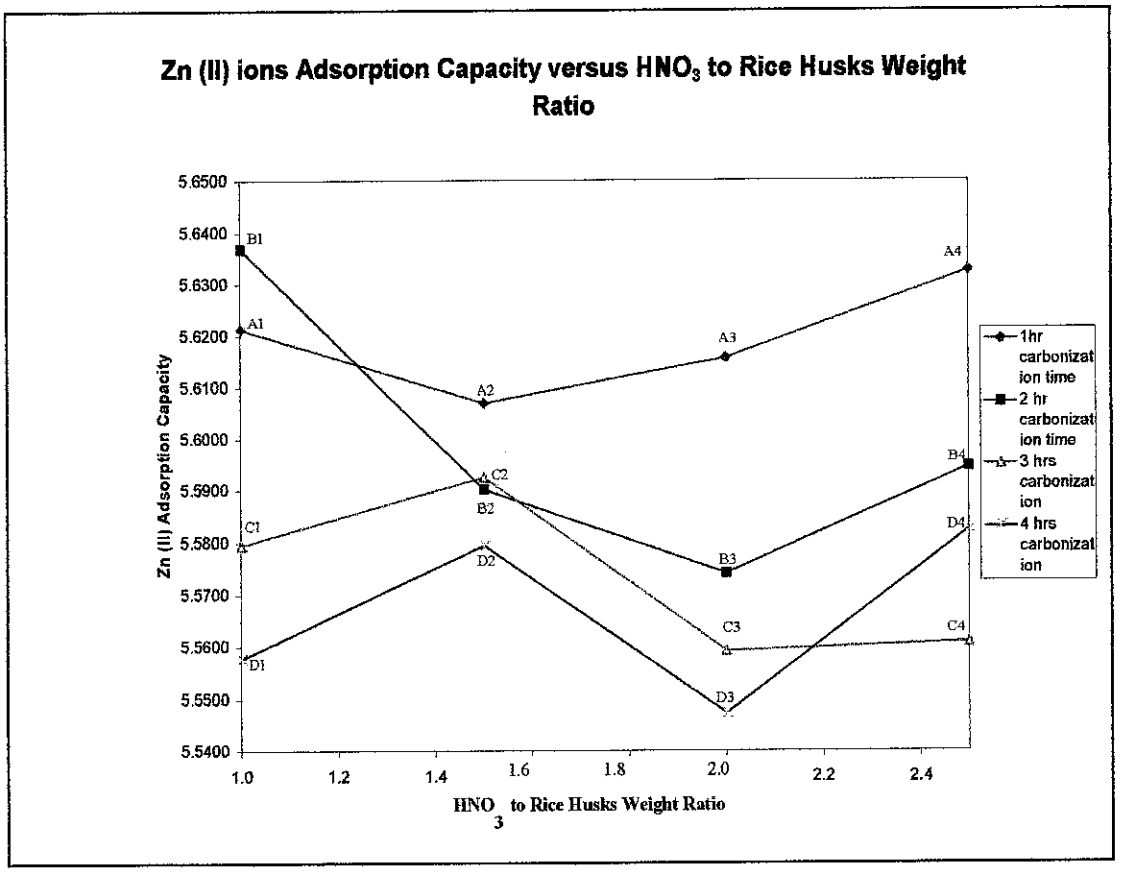

Figure 15: Graph of Zn(II) Ion Adsorption Capacity vs. Nitric Acid to Rice Husks Weight Ratio

Castro et. al reported that for a specific heat treatment temperature, increasing the weight ratio of phosphoric acid (activating agent) will increase the S_{BET} to a maximum value before decreasing with the continuing increment of the weight ratio. The same goes to the other surface properties such as total volume, microporous volume, mesoporous volume and mean pore radius. The maximum value of S_{BET} for the works of Castro et. al. using sugar cane bagasses subjected to heat treatment temperature of 500°C occurred at the weight ratio of 1.5:1.0.

In the author's case, the maximum adsorption capacity was achieved at weight ratio of 1:1 and at heat treatment of 2 hours that of Sample B1. Hence, Sample B1 will be used in the studies of loading size effect and contact time effect in Section 4.3.5 and Section 4.3.6, respectively.

4.3.4 Effect of Nitric Acid to Rice Husks Weight Ratio on Cu(II) Adsorption

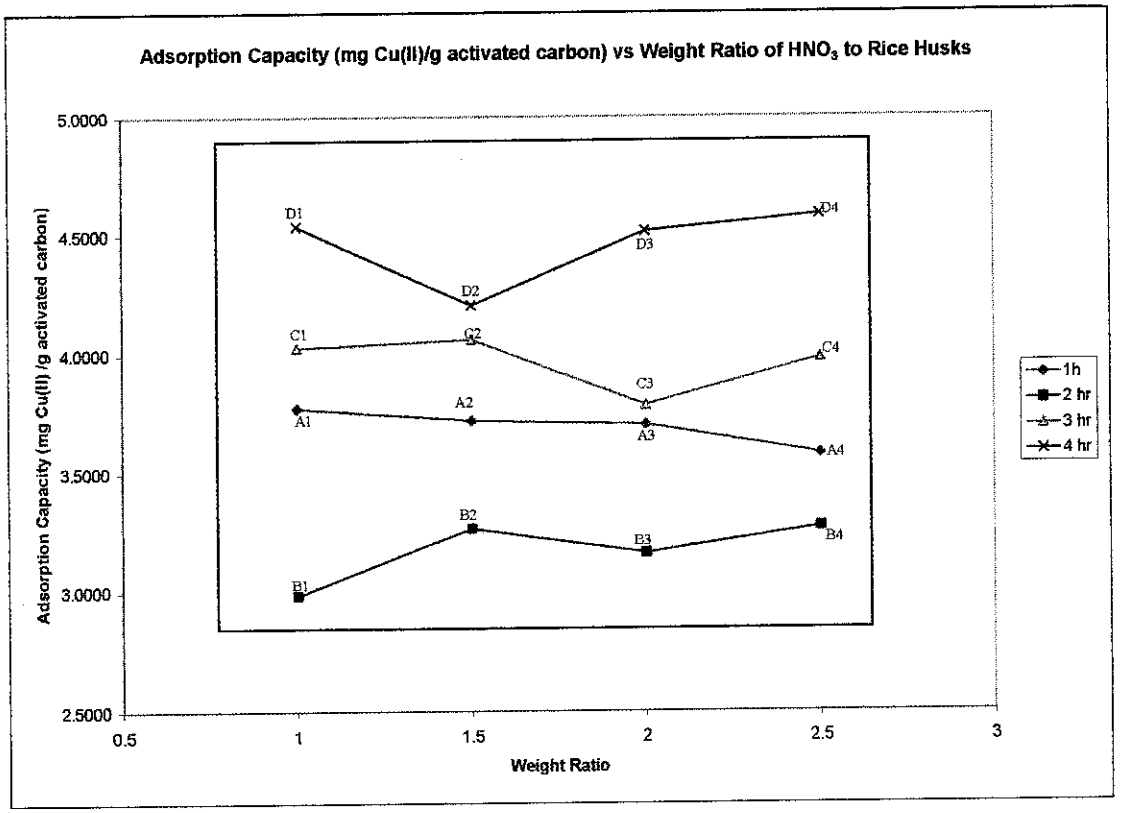

Figure 16: Graph of Copper Adsorption Capacity vs. Nitric Acid to Rice husks Weight Ratio

Increment of HNO_3 to rice husks weight ratio was only significant in the case of 2hr heat treatment. However, in magnitude, the highest adsorption capacity was achieved by the sample that was pyrolysed for 4 hours and at weight ratio of 2.5:1.0. This also shows that at high temperature treatment, the high weight ratio may also increase the adsorption capacity.

Lastly from the graph above, one can see the ranking of which carbonization time is ideal for processing activated carbons to adsorb Copper(II) ions. From the highest ranking to the lowest, the appropriate carbonization durations are 4 hours, 3 hours, 1

hour and 2 hours. From the combination of weight ratio and carbonization aspect, the activated carbon sample that produces highest adsorption capacity for Copper(II) is Sample D4 which will be used in the studies on effect of loading size and contact time in Section 4.3.5 and Section 4.3.6, respectively.

4.3.5 Effect of Sample Loading on Zn (II) and Cu (II) Adsorption

Table 15: Loading Size and Amount Zn (II) Removal

Sample Weight	Residue concentration of Zn(II) Ions (mg/L)	Initial concentration of Zn(II) Ion (mg/L)	Amount of Zn (II) adsorbed (mg)	Adsorption capacity (mg Zn (II) ions adsorbed/g carbon)	Percent Removal of Zn(II) Ins
0.1001	2.1300	24.7	0.56425	5.63686	91.37652
0.2001	0.3655	24.7	0.60836	3.04029	98.52024
0.3001	0.1994	24.7	0.61251	2.04103	99.19271
0.4000	0.0033	24.7	0.61741	1.54354	99.98656

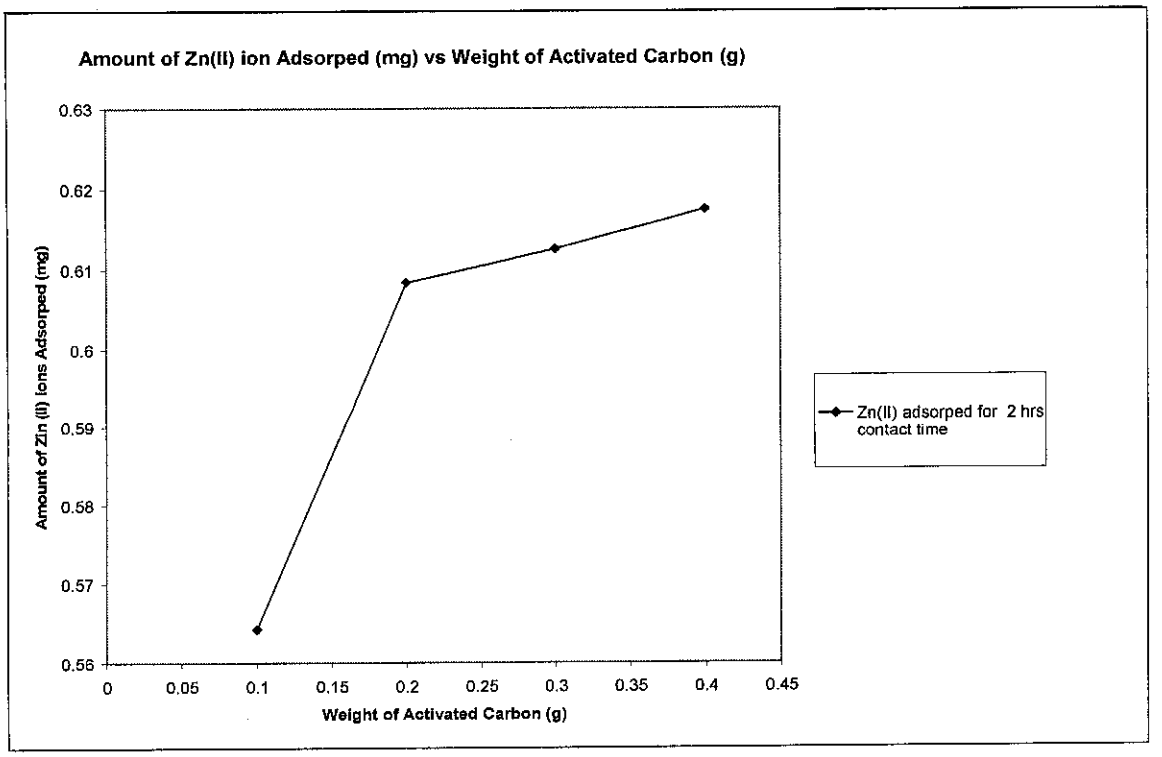

Figure 17: Graph of Amount of Zn (II) Removed Vs Loading Size

From the Table 15 and Figure17, it can be concluded that a higher loading can increase the removal of Zn (II) ions. However, the adsorption capacity of activated carbon has been lowered slightly with the increase of loading size.

Table 16: Loading Size and Amount Cu(II) Removed

Sample Weight (g)	Residue concentration of Cu(II) Ions (mg/L)	Initial concentration of Cu(II) Ion (mg/L)	Amount of Cu (II) adsorbed (mg)	Adsorption capacity (mg Zn (II) ions adsorbed/g carbon)	Percent Removal
0.1003	6.610	25.00	0.4598	4.5837	73.56
0.2000	2.586	25.01	0.5606	2.8030	89.65
0.3000	0.532	25.01	0.6120	2.0399	97.87
0.4000	0.083	25.01	0.6232	1.5579	99.66

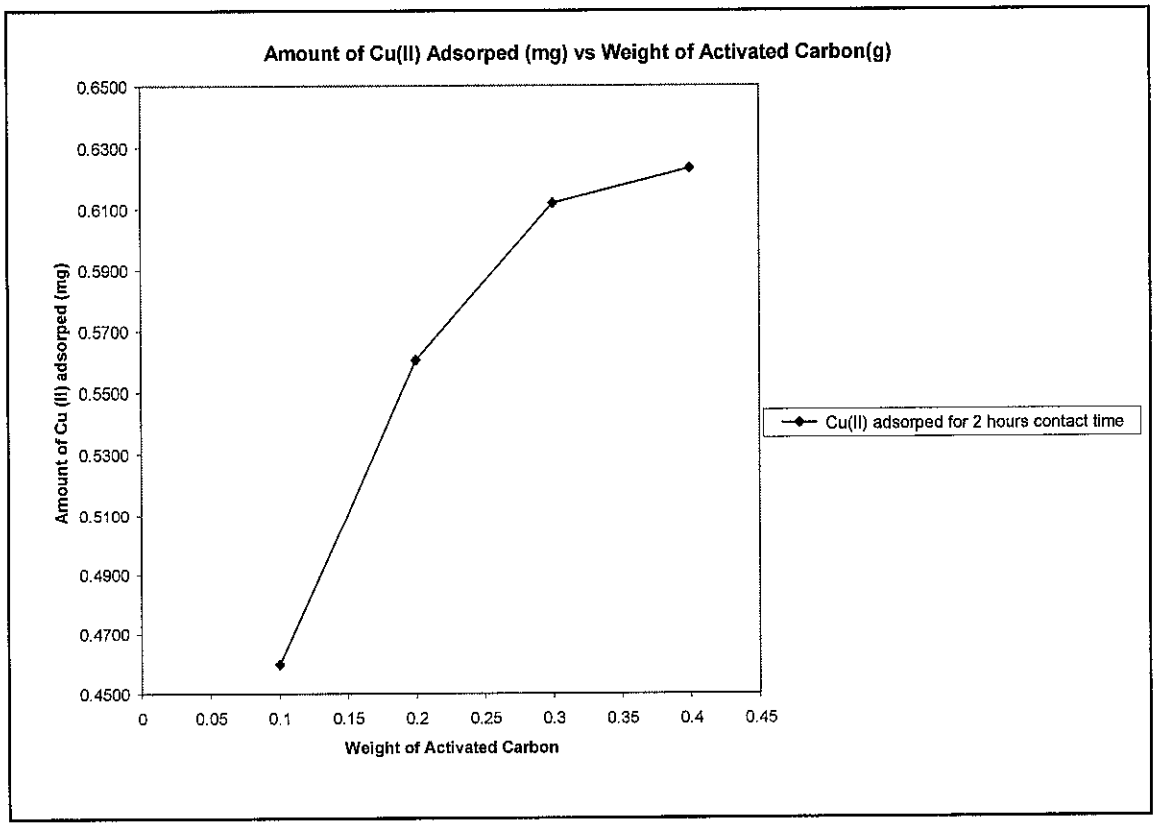

Figure 18: Graph of Amount of Cu(II) Removed vs. Loading Size

The loading effect of the sample is significant. Using 0.4 g instead of 0.1 g of activated carbon can actually increase the percent removal from 70% to 99%. This happens because, when more activated carbons are used, the number of adsorption sites increase, hence resulting in more metal ions being adsorbed.

4.3.6 Effect of Adsorption Contact Time

4.3.6.1 Zn(II)Adsorption

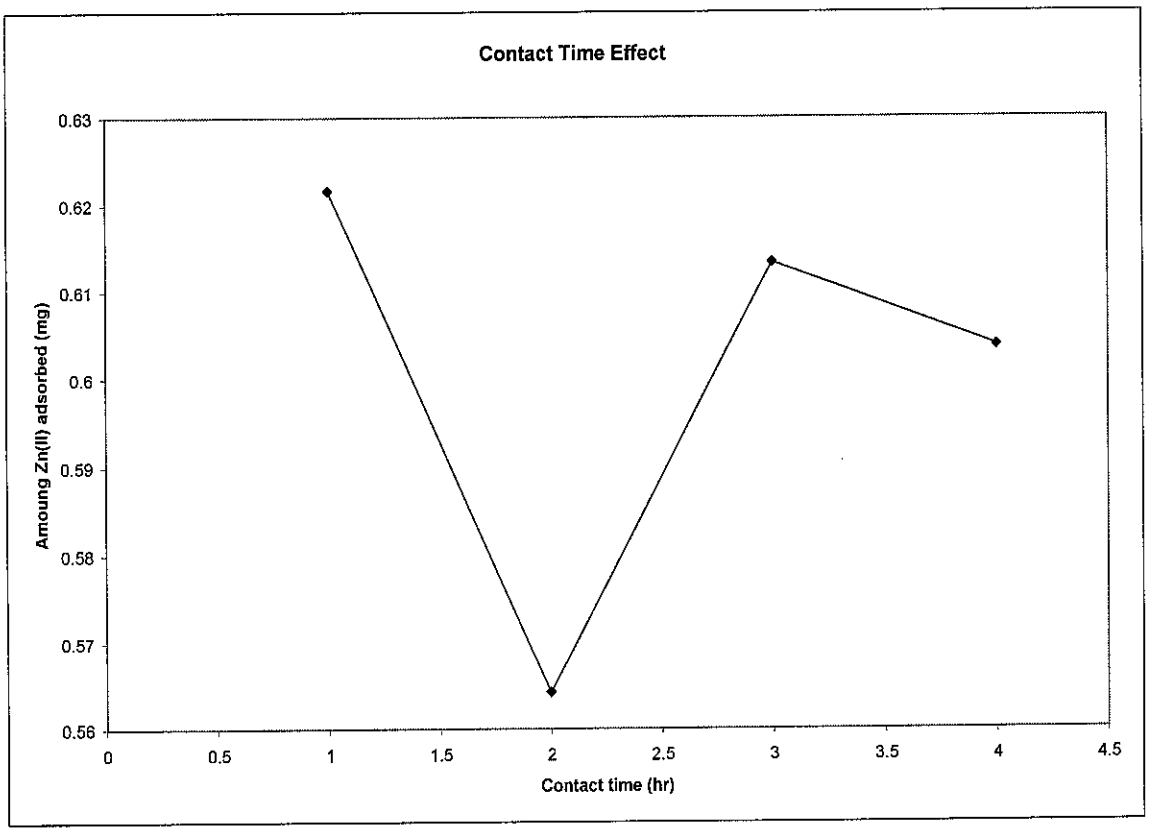

Figure 19: Amount of Zn(II) Ions Adsorbed vs. Contact Time

Contact time is the duration of which the activated carbon is contacted with the solution. In the author's experiment, this meant the duration of which the samples are left to shake on the shaker machine for a set duration of 1, 2, 3 and 4 hours.

For Zn (II) adsorption process, the highest amount of Zn (II) ions was adsorbed on the 1st hour. The lowest point was on the second hour. The reason for the drop of adsorption during the second hour could be due to desorption from the rice husks itself.

4.3.6.2 Cu(II)Adsorption

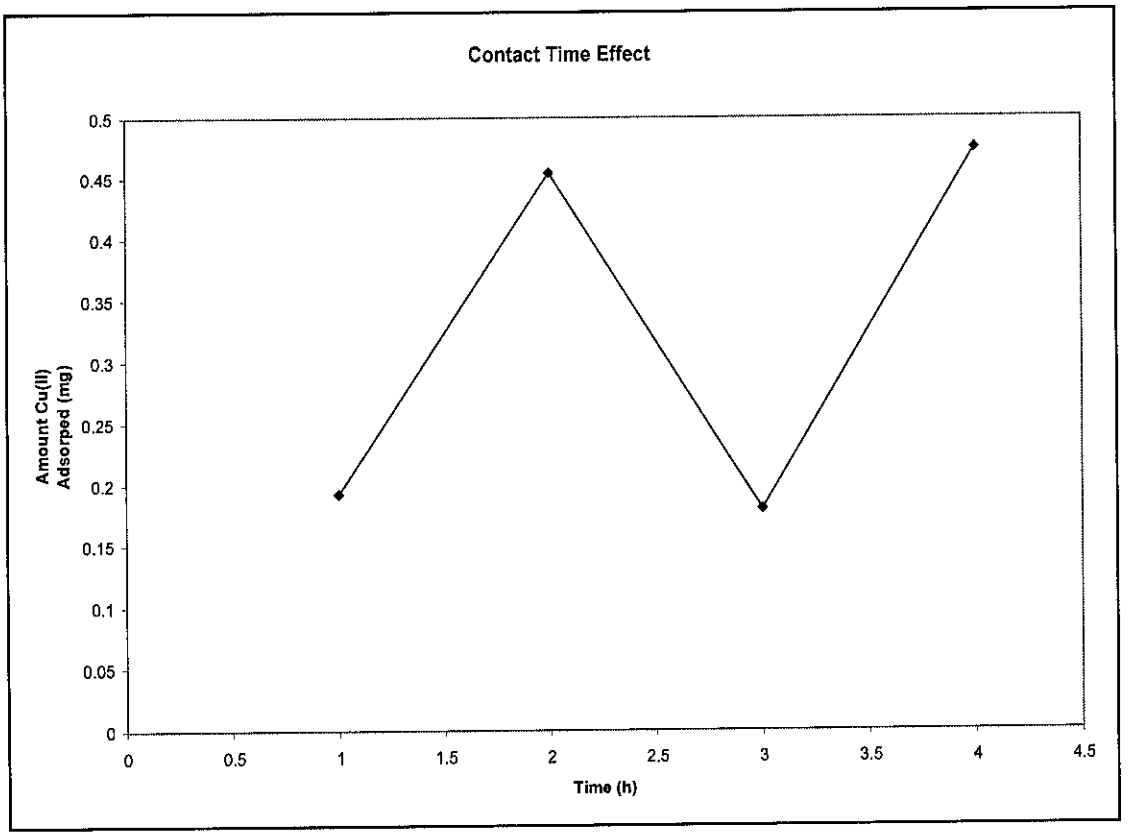

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Figure 20: Amount of Cu (II) Ions Adsorbed vs. Contact Time

The contact time effect for Cu(II) adsorption is just the opposite with that of the Zn (II) adsorption. The highest adsorption was on the fourth hour.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Activated carbons produced from rice husks digested in nitric acid and pyrolysed at 500°C can indeed be used for the removal of Zn(II) ions and Cu(II) ions in industrial wastewater. From the research done, it was found that 0.1g of the activated carbon could actually achieve 73.6% removal of copper (II) ions and 91.38% of Zn(II) ions. Used in larger quantities, these activated carbons could even achieve up to 99 % Cu(II) and Zn (II).

Upon digestion in nitric acid 3M, some weight of the raw rice husks was lost. Carbonization or pyrolysis conducted in a muffle furnace also caused more weight loss to the sample. Hence the activated carbon yield was not very high. However, the sample with the highest yield is Sample B1, digested with weight ratio of 1:1 and pyrolysed at 500°C for 2 hours.

Through the experimental works done on the rice husks activated carbon, it was concluded that carbonization time, weight ratio of nitric acid to rice husks, loading size and contact time do have influences on the yield and the performance of the activated carbon. For the adsorption of Zn(II) ions, the best sample identified was B1, pyrolised for 2 hours at 1:1 acid to rice husks weight ratio, where as the best sample for Cu(II) adsorption was D4 , pyrolysed for 4 hours at weight ratio 2.5:1. On the whole these activated carbons are more suitable for the removal of zinc ions compared to the copper ions.

It is also concluded that as the weight ratio of nitric acid to rice husks increase, there will be an increase in the adsorption capacity of the activated carbon prepared. However, this increment is limited to a maximum value whereby any additional increment of the weight ratio will not increase the adsorption capacity. In terms of carbonization time, an increment of carbonization time would increase the adsorption capacity. Once the maximum adsorption capacity has been achieved, continual increment of carbonization time could in fact lower the adsorption capacity. This explains why in some cases, samples carbonized for shorter duration tend to be more effective than those carbonized for a few more hours.

The contact time between the activated carbons and the ionic solutions may also influence the amount of metal ions being adsorbed. If the contact time is too short, the amount of ions adsorbed may not have reached its full capacity. On the other hand, if the contact time is too long, desorption may occur and this will result in no significant change in the initial concentration of the solution.

5.2 Recommendations

As the research project has been completed, several recommendations have been identified to aid in improving the current research for future use.

First, it is recommended that a larger range of carbonization time should be tested in producing the activated carbon. The range should begin with 0 hr and increase in intervals of 30 minutes. This way, the researcher can identify the maximum carbonization time before the adsorption capacity changes negatively.

On the issue of equipment, it would be better to run the pyrolysis in a furnace that is equipped with inert air flow such as argon flow. This would ensure that no oxygen could come in contact with the rice husks, hence preventing the formation of ashes.

In addition, each sample should be analyzed on its surface area, total pore volume, micro porous volume, mesoporous volume and also the mean pore radii. This will provide more solid evidence on the effects of weight ratio, carbonization time and the combination of those two factors on the surface area development.

For a better study on the effect of adsorption contact time, the experiment should be run for a longer period. This would provide sufficient adsorption data to determine the equilibrium time of the activated carbon.

Finally, further works should be done to determine the reason why the adsorption capacity of the rice husks activated carbons towards Zn(II) ions is better than that of Cu(II) ions. Indeed, these recommendations should be taken into consideration for future research to enrich the current findings on this particular subject.

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APPENDICES

APPENDIX I: Instructions for Preparation of Reagents

The following procedures were used in preparing the nitric acid 3M, 25 ppm Zn(II) Nitrate solution and 25 ppm Cu(II) Nitrate solution.

Nitric Acid Preparation

In preparing dilute acid, concentrated acid should be added to water, not vice versa. Nitric acid of 3 M was prepared following the steps below:

i) Fill a 1000 mL beaker with 200 mL distil water

ii) Place the beaker on a magnetic stirrer and let it stir at the lowest speed.

iii) Add 208 mL of 14.4 M Nitric Acid by slowly pouring it into the beaker

iv) Let the mixture stir for 5 minutes and transfer the entire contents of the beaker into a 1000 mL volumetric flask.

v) Add distil water up to the mark of the volumetric flask.

vi) Cap the volumetric flask and shake the flask to mix the solution evenly.

Calculations for Preparation of Nitric Acid 3 M

The dilution formula is the following:

$$M_1V_1 = M_2V_2$$

Where M_1 = Initial concentration of acid

M_2= Desired concentration of acid

V_1= Required volume of acid of concentration M1

V_2 = Desired volume of acid after dilution [Eq. A1]

Given the following data:

Purity= 65%

Density = 1.40g/ mL

Molecular weight = 63.01g/ mole

$$M_1 = (\text{Density} \times \text{Purity}) / \text{Molecular Weight} \quad \text{[Eq. A2]}$$

Therefore,

M_1 = [1.40g/ mL x (1000mL/1L) x 0.65] / 63.01 g/ mole

= 14.4 mole/L

= 14.4 M

Substituting into [Eq A.1],

$V1 = (M_2V_2)/M_1$

= (3M x 1L)/ 14.4M

= 0.208 L

Hence, 208 mL nitric acid 65% is needed for dilution to prepare 1000mL of nitric acid

Zinc(II) Nitrate Solution Preparation

The following steps are done to prepare 1000 mL Zinc (II) Nitrate solution of 25ppm

i) Fill a 1000 mL beaker with 400 mL distil water

ii) Place the beaker on a magnetic stirrer and let it stir at the lowest speed.

iii) Add 113.7 mg. of $Zn(NO_3)_2.6H_2O$ hydrated into the beaker

iv) Let the mixture stir for 10 minutes to allow the solids to dissolve and transfer the entire contents of the beaker into a 1000 mL volumetric flask.

v) Add distil water up to the mark of the volumetric flask. Cap the volumetric flask and shake the flask to ensure proper mixing

Calculations To Prepare 1000mL 25 ppm Zn^{2+} ions

Formula Weight of Zn ion = 65.39g/mol

Formula Weight of $Zn(NO_3)_2.6H_2O$ = 297.47 g/mole

25 ppm = 25mg/ L

Moles of Zn^{2+} required = $25 \times 10^{-3} \times 1$ mol/ 65.39g

$= 3.823 \times 10^{-4}$ moles

To obtain 3.823×10^{-4} moles of Zn^{2+} ions, we require 3.823×10^{-4} moles of $Zn(NO_3)_2.6H_2O$.

$\Rightarrow$ Weight of $Zn(NO_3)_2.6H_2O$ required $= 3.823 \times 10^{-4}$ moles x 297.47g/mol

$= 0.1137$ g

$= 113.7$ mg.

Hence 113.7 mg of $Zn(NO_3)_2.6H_2O$ is needed to prepare 1000mL of 25 ppm Zn^{2+} ions.

Copper (II) Nitrate Solution Preparation

All the steps for preparing Zinc (II) Nitrate Solution were carried out by replacing the $Zn(NO_3)_2.6H_2O$ hydrated with 95.05 mg $Cu(NO_3)_2.3H_2O$ hydrate.

Calculations To Prepare 1000mL 25 ppm Cu^{2+} ions

Formula Weight of Cu ion = 63.546 g/mol

Formula Weight of $Cu(NO_3)_2.3H_2O$ = 241.60 g/mole

25 ppm = 25mg/ L

Moles of Zn2+ required = $25 \times 10^{-3} \times 1$ mol/ 63.546 g

$= 3.934 \times 10^{-4}$ moles

To obtain 3.934×10^{-4} moles of Cu^{2+} ions, we require 3.934×10^{-4} moles of $Cu(NO_3)_2.3H_2O$.

$\Rightarrow$ Weight of $Cu(NO_3)_2.3H_2O$ required $= 3.934 \times 10^{-4}$ moles x 241.60 g/mol

$= 0.0950$ g

$= 95.05$ mg.

Hence 95.05 mg of $Cu(NO_3)_2.3H_2O$ is needed to prepare 1000mL of 25 ppm Cu^{2+} ions.

APPENDIX II: Results of Activated Carbon Preparation

Table A.II. 1: Summary of experimental data to calculate the weight loss after acid digestion during first trial of preparation

Sample Name	HNO_3 to Rice Husks Weight Ratio	Weight of Rice husks (g)	Volume of Nitric Acid (mL)	Weight of crucible (g)	Weight of Crucible + Rice Husks After Digestion (g)	Weight of Digested Rice husks (g)	Percentage Weight loss %
A1	1.0:1.0	1.00	5.29	33.24	34.15	0.91	9.00
B1	1.0:1.0	1.01	5.34	35.76	36.68	0.93	8.90
C1	1.0:1.0	1.01	5.34	32.77	33.68	0.91	9.90
A2	1.5:1.5	1.00	7.94	32.69	33.62	0.93	7.00
B2	1.5:1.5	1.01	8.01	34.24	35.17	0.94	7.90
C2	1.5:1.5	1.01	8.01	33.32	34.254	0.93	7.60
A3	2.0:1.0	1.00	10.58	37.36	38.292	0.93	6.80
B3	2.0:1.0	1.01	10.69	32.048	32.988	0.95	6.90
C3	2.0:1.0	1.01	10.69	37.45	38.409	0.96	5.05
A4	2.5:1.0	1.00	13.23	34.78	35.68	0.90	10.00
B4	2.5:1.0	1.01	13.36	28.28	29.09	0.91	9.90
C4	2.5:1.0	1.01	13.36	34.85	35.748	0.90	11.10

Note: Volume of nitric acid can be calculated using the following equation:

$$\textit{Volume of Nitric Acid} = \textit{(Weight Ratio)} \times \textit{(1 mole/63.01 g)} \times \textit{(1000mL /3 moles)} \quad \text{[Eq. A.3]}$$

Table A.II. 2: Summary of experimental data to calculate the activated carbon yield for first trial

Sample Name	Weight Ratio	Weight of Rice husks (g)	Weight of crucible (g)	Weight of Crucible + Rice Husks After Carbonization (g)	Weight of Activated Carbon (g)	Activated Carbon Yield (%)
A1	1.0:1.0	1.00	33.24	33.402	0.162	16.2
A2	1.5:1.0	1.00	35.76	32.846	0.156	15.6
A3	2.0:1.0	1.00	32.77	37.68	0.320	32.0
A4	2.5:1.5	1.00	32.69	34.925	0.145	14.5
B1	1.0:1.5	1.01	34.24	35.82	0.060	5.94
B2	1.5:1.5	1.01	33.32	34.25	0.010	0.99
B3	2.0:1.0	1.01	37.36	32.077	0.029	2.87
B4	2.5:1.0	1.01	32.048	28.35	0.070	6.93
C1	1.0:1.0	1.01	37.45	32.81	0.040	3.96
C2	1.5:1.0	1.01	34.78	33.37	0.050	4.95
C3	2.0:1.0	1.01	28.28	37.49	0.040	3.96
C4	2.5:1.0	1.01	34.85	34.89	0.040	3.96

Table A.II.3: Summary of experimental data to calculate the percentage weight loss of rice husks after digestion for final activated carbon preparation

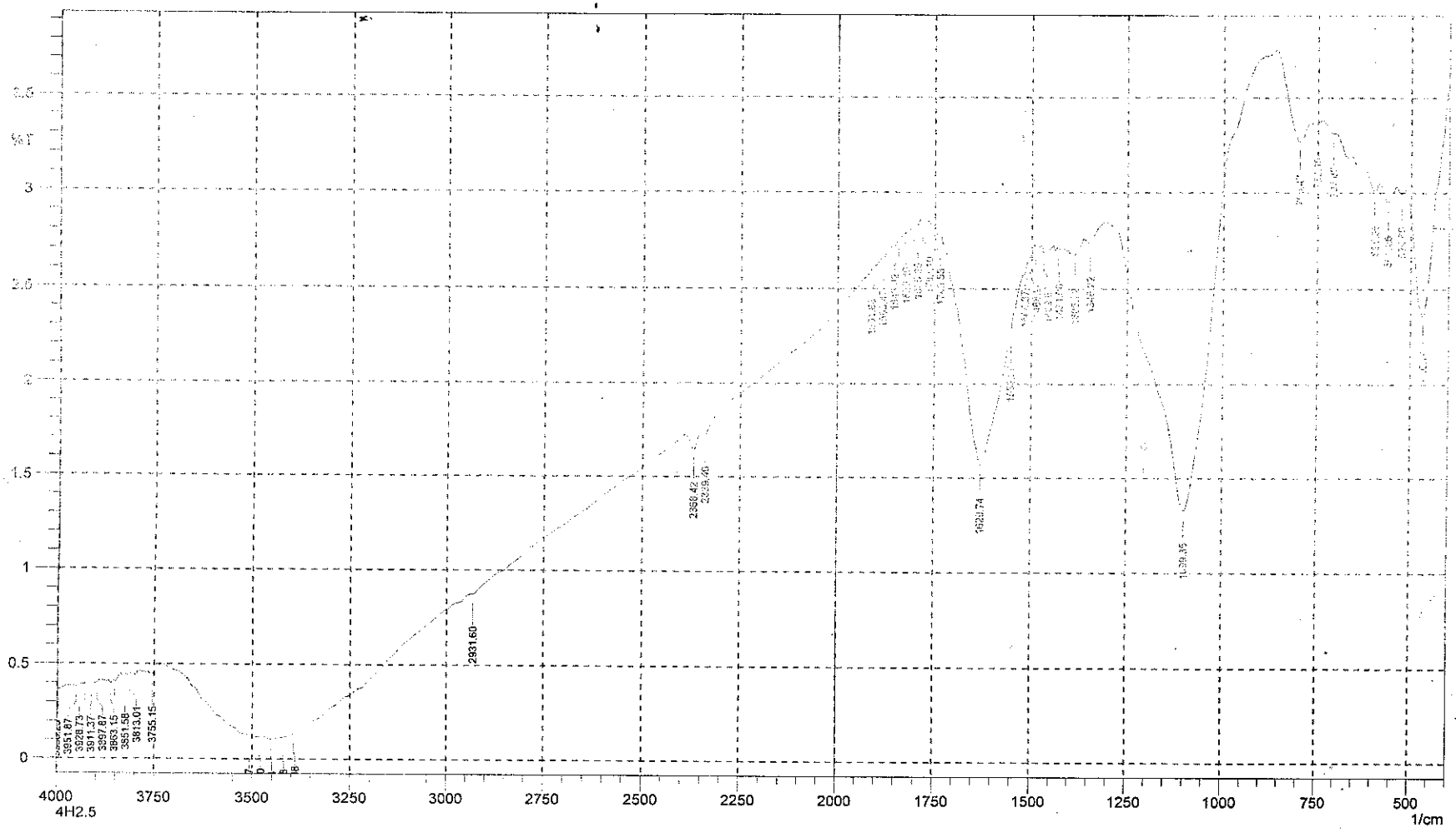
Sample Name	HNO_3 to Rice Husks Weight Ratio	Weight of Rice Husks Initially (g)	Weight of Crucible (g)	Weight of Crucible + Rice Husks After Digestion (g)	Weight of Digested Rice husks (g)	Percentage Weight Loss After HNO_3 Digestion (%)
A1	1.0:1.0	10	56.1	65.15	9.05	9.5
A2	1.5:1.0	10	61.61	70.7	9.09	9.1
A3	2.0:1.0	10	55.2	64.21	9.01	9.9
A4	2.5:1.0	10	52.78	61.74	8.96	10.4
B1	1.0:1.0	10	56.10	65.31	9.21	7.86
B2	1.5:1.0	10	61.62	70.69	9.07	9.3
B3	2.0:1.0	10	55.18	64.41	9.23	7.7
B4	2.5:1.0	10	52.79	61.89	9.10	9
C1	1.0:1.0	10	56.10	65.61	9.51	4.9
C2	1.5:1.0	10	61.62	71.14	9.52	4.8
C3	2.0:1.0	10	55.18	64.55	9.37	6.3
C4	2.5:1.0	10	52.79	62.38	9.41	5.9
D1	1.0:1.0	10	114.74	123.7	8.96	10.4
D2	1.5:1.0	10	133.14	142.08	8.94	10.6
D3	2.0:1.0	10	111.65	120.51	8.86	11.4
D4	2.5:1.0	10	108.48	117.62	8.82	11.8

Table A.II.4: Summary of experimental data to calculate the activated carbon yield for final activated carbon preparation

Sample	Initial Weight of rice husks (g)	Weight of Crucible (g)	Weight of Crucible + Rice Husks After Carbonization (g)	Weight of Activated Carbon (g)	Activated Carbon Yield (%)
A1	10	56.1	59.56	3.46	34.60
A2	10	61.61	64.98	3.37	33.70
A3	10	55.2	58.50	3.30	33.00
A4	10	52.78	55.99	3.21	32.10
B1	10	56.10	59.88	3.78	37.77
B2	10	61.62	64.88	3.26	32.57
B3	10	55.18	58.33	3.15	31.48
B4	10	52.79	56.07	3.28	32.78
C1	10	56.10	59.15	3.05	30.50
C2	10	61.62	58.35	2.96	29.60
C3	10	55.18	64.58	3.17	31.70
C4	10	52.79	55.69	2.9	29.00
D1	10	114.74	107.01	2.8	28.00
D2	10	133.14	94.67	2.928	29.28
D3	10	111.65	104.25	2.94	29.4
D4	10	108.48	103.95	2.87	28.7

APPENDIX III: FTIR Spectrum for Sample D4 of Activated Carbon

SHIMADZU



Comment;
4H2.5

No. of Scans;
Resolution;
Apodization;

Date/Time; 05/09/2005 03:32:30 PM
User; Organic chemistry

APPENDIX IV: Results and Data to Calculate Adsorption Capacities

In this section, the main variable of interest is the adsorption capacity of each activated carbon sample prepared under different combination of carbonization time and nitric acid to rice husks weight ratio. The data for adsorption capacities of Zinc(II) ions and Copper(II) ions are tabulated in Table A.IV.1 Table A.IV.2 respectively. Each sample was tested with 25 mL of $Zn(NO_3)_2$ and $Cu(NO_3)_2$ aqueous solutions.

Table A.IV.1: Experimental data for adsorption of Zinc(II) ions for contact time of 2 hours

Sample Name	Carbonization Time(hr)	Weight Ratio Of HNO_3 To Rice Husks (g/g)	Sample Weight (g)	Initial concentration of Zn(II) Ion (mg/L)	Residue concentration of Zn(II) Ions (mg/L)	Amount of Zn (II) adsorbed (g)	Adsorption capacity (mg Zn (II) ions adsorbed/ g carbon)
A1	1	1.0	0.1002	24.7	2.17	0.5633	5.6213
A2	1	1.5	0.1005	24.7	2.16	0.5635	5.6070
A3	1	2.0	0.1003	24.7	2.17	0.5633	5.6157
A4	1	2.5	0.1000	24.7	2.17	0.5633	5.6325
B1	2	1.0	0.1001	24.7	2.13	0.5643	5.6369
B2	2	1.5	0.1004	24.7	2.25	0.5613	5.5901
B3	2	2.0	0.1006	24.7	2.27	0.5608	5.5741
B4	2	2.5	0.1005	24.7	2.21	0.5623	5.5945
C1	3	1.0	0.1001	24.7	2.36	0.5585	5.5794
C2	3	1.5	0.1000	24.7	2.33	0.5593	5.5925
C3	3	2.0	0.1006	24.7	2.33	0.5593	5.5591
C4	3	2.5	0.1003	24.7	2.39	0.5578	5.5608
D1	4	1.0	0.1004	24.7	2.38	0.5580	5.5578
D2	4	1.5	0.1001	24.7	2.36	0.5585	5.5794
D3	4	2.0	0.1005	24.7	2.40	0.5575	5.5473
D4	4	2.5	0.1000	24.7	2.37	0.5583	5.5825

The following equations were used to calculate the adsorption capacity for Zn(II) ions:

$$\textit{Amount of Zn(II) ion adsorbed} = \frac{25}{1000}(\textit{Initial Concentration-Residue Concentration}) \quad \text{[Eq. A.4]}$$

$$\textit{Adsorption Capacity} = \frac{\textit{Amount of Zn (II) ion adsorbed}}{\textit{Sample weight}} \quad \text{[Eq. A.5]}$$

Table A.IV.2: Experimental data for adsorption of Copper(II) ions for contact time of 2 hours

Sample Name	Carbonization Time(hr)	Weight Ratio Of HNO_3 To Rice Husks (g/g)	Sample Weight (g)	Initial concentration of Cu(II) Ion (mg/L)	Residue concentration of Cu(II) Ions (mg/L)	Amount of Cu(II) adsorbed (g)	Adsorption capacity (mg Cu(II) ions adsorbed/ g carbon)
A1	1	1.0	0.1001	25	9.88	0.3780	3.7762
A2	1	1.5	0.1000	25	10.11	0.3723	3.7225
A3	1	2.0	0.1004	25	10.12	0.3720	3.7052
A4	1	2.5	0.1000	25	10.67	0.3583	3.5825
B1	2	1.0	0.1002	25	13.03	0.2993	2.9865
B2	2	1.5	0.1000	25	11.94	0.3265	3.2650
B3	2	2.0	0.1002	25	12.33	0.3168	3.1612
B4	2	2.5	0.1004	25	11.86	0.3285	3.2719
C1	3	1.0	0.1005	25	8.8	0.4050	4.0299
C2	3	1.5	0.1002	25	8.72	0.4070	4.0619
C3	3	2.0	0.1003	25	9.81	0.3798	3.7861
C4	3	2.5	0.1004	25	9	0.4000	3.9841
D1	4	1.0	0.1001	25	6.83	0.4543	4.5380
D2	4	1.5	0.1002	25	8.14	0.4215	4.2066
D3	4	2.0	0.1002	25	6.91	0.4523	4.5135
D4	4	2.5	0.1003	25	6.61	0.4598	4.5837

The following equations were used to calculate the adsorption capacity for Cu(II) ions:

$$\textit{Amount of Cu(II) ion adsorbed} = \frac{25}{1000}(\textit{Initial Concentration-Residue Concentration}) \quad \text{[Eq. A.6]}$$

$$\textit{Adsorption Capacity} = \frac{\textit{Amount of Cu (II) ion adsorbed}}{\textit{Sample weight}} \quad \text{[Eq. A.7]}$$

APPENDIX V: Experimental Data and Calculations for Study on Effect of Activated Carbon Loading Size

Table A.V.1: Data for Effect of Loading Size on the Adsorption of Zinc (II) Ions

Sample Weight	Residue concentration of Zn(II) Ions (mg/L)	Initial concentration of Zn(II) Ion (mg/L)	Amount of Zn (II) adsorbed (mg)	Adsorption capacity (mg Zn (II) ions adsorbed/g carbon)	Percent Removal of Zn(II) Ins
0.1001	2.1300	24.7	0.56425	5.63686	91.37652
0.2001	0.3655	24.7	0.60836	3.04029	98.52024
0.3001	0.1994	24.7	0.61251	2.04103	99.19271
0.4000	0.0033	24.7	0.61741	1.54354	99.98656

The table above presents the data obtained for activated carbon of Sample B1.The adsorption capacity, amount of ions adsorbed were calculated using the equations in APPENDIX IV. The percent of Zn (II) removal can be calculated using the equation below:

$$\text{Percent of Zn(II) Removal} = \frac{[\text{Initial Concentration of Zn(II)} - \text{Residue Concentration of Zn(II)}]}{\text{Initial Concentration of Zn(II)}} \times 100\% \quad \text{[Eq. A.8]}$$

Table A.V. 2: Data for Effect of Loading Size on The Adsorption of Copper(II) Ions

Sample Weight	Residue concentration of Cu(II) Ions (mg/L)	Initial concentration of Cu(II) Ion (mg/L)	Amount of Cu (II) adsorbed (mg)	Adsorption capacity (mg Zn (II) ions adsorbed/g carbon)	Percent Removal
0.1003	6.61	25.00	0.4598	4.58374875	73.56
0.2000	2.5862	25.01	0.560595	2.802975	89.65
0.3000	0.5316	25.01	0.61196	2.03986667	97.87
0.4000	0.0833	25.01	0.623168	1.55791875	99.66

The table above tabulates the data obtained for the activated carbon sample D4. Sample D4 was used for this experiment because of its highest performance in adsorbing Cu(II) in the first batch of adsorption tests. The percentage of Cu(II) ions removal can be calculated as shown below:

$$\text{Percent Removal Of Cu(II)} = \frac{[\text{Initial Concentration of Zn(II)} - \text{Residue Concentration of Cu(II)}]}{\text{Initial Concentration of Cu(II)}} \times 100\% \quad \text{[Eq. A.9]}$$

APPENDIX VI: Experimental Data for Effect of Contact Time on Adsorption Capacity

Table A.VI.1 Data for Adsorption of Zn(II) Ions Under Varying Contact time

Contact Time (hr)	Sample Weight	Initial Concentration of Zn(II) Ion (mg/L)	Residue Concentration of Zn(II) Ions (mg/L)	Amount of Zn(II) Adsorbed (mg)	Adsorption Capacity (mg Zn (II) Ions Adsorbed/g Carbon)	Percent of Zn(II) Ions Adsorbed (%)
1	0.1001	24.7	0.1329	0.6142	6.1356	99.46
2	0.1001	24.7	2.1300	0.5643	5.6369	91.38
3	0.1001	24.7	0.4652	0.6059	6.0526	98.12
4	0.1001	24.7	0.6492	0.6013	6.0067	97.37

The table above shows the data obtained for adsorption of Zn(II) ions onto Sample B1 at different carbonization time of 1, 2, 3 and 4 hours. Sample B1 is the activated carbon pyrolysed at 500°C for 2 hours and its nitric acid to rice husks weight ratio is 1.5:1.The amount of Zn (II) adsorbed, the adsorption capacity and percentage of ions adsorbed can be calculated by using the Equation A.4, Equation A.5 and Equation A.8 respectively.

Table A.VI.2 Data for Adsorption of Cu(II) Ions Under Varying Contact time

Contact Time (hr)	Sample Weight	Initial concentration of Cu(II) Ion (mg/L)	Residue concentration of Cu(II) Ions (mg/L)	Amount of Cu (II) adsorbed (mg)	Adsorption capacity (mg Cu (II) ions adsorbed/g carbon)	Percent of Cu(II) ions adsorbed (%)
1	0.1000	25	17.3127	0.192183	1.921825	30.7492
2	0.1000	25	6.83	0.4543	4.52891326	72.68
3	0.1000	25	17.805	0.1799	1.79875	28.78
4	0.1000	25	6.03	0.4743	4.7425	75.88

Table A.VI.2 shows the data obtained for adsorption of Cu(II) ions onto Sample D4 at different carbonization time of 1, 2, 3 and 4 hours. Sample D4 is the activated carbon pyrolysed at 500°C for 2 hours and its nitric acid to rice husks weight ratio is 2.5:1.The amount of Cu(II) adsorbed, the adsorption capacity and percentage of ions adsorbed can be calculated by using the Equation A.6, Equation A.7 and Equation A.9 respectively.

APPENDIX VII: Results from AAS

The following pages are the printouts obtained from the Atomic Absorption Spectrophotometer. The concentration of Zn(II) ions and Cu(II) ions in these printouts refer to the residue concentration of these metal ions in the sample. Residue concentration is the concentration of the ion after adsorption by the activated carbon prepared.

Detail Table 4/08/05 11:33PM

Analysis Name : Fairene Leong
Comment : Zn (II) Adsorption , 2 hrs
Meas. Date : 4/08/05 11:33 AM
Element : Zn

Sample ID	STD1	Sample Name	ABS	REF
No.	Corr. Conc (ppm)	Conc. (ppm)	0.0001	-0.0010
1	--	0.00	0.0001	-0.0010
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 2	Sample Name	ABS	REF
No	Corr. Conc (ppm)	Conc. (ppm)	0.0832	0.0166
1	--	0.20	0.0539	0.0345
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 3	Sample Name	ABS	REF
No	Corr. Conc	Conc. (ppm)	0.1581	0.0262
1	--	0.40	0.1581	0.0262
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 4	Sample Name	ABS	REF
No	Corr. Conc (ppm)	Conc. (ppm)	0.2968	0.0452
1	--	0.80	0.2968	0.0452
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Coefficient : K3=--
K2=--
K1=3.684285E-001
K0=5.600001E-003

Corr Coef : 0.9991

Sample ID	UNK- 001	Sample Name:A1		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8037	0.5563
Mean	2.18	2.18	0.8037	0.5563
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 002	Sample Name: A2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8075	0.5537
Mean	2.18	2.18	0.8075	0.5537
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 003	Sample Name: A3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8075	0.5507
Mean	2.18	2.18	0.8075	0.5507
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 004	Sample Name:A4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.19	2.19	0.8120	0.5459
Mean	2.19	2.19	0.8120	0.5459
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00
	6.03			

Sample ID	UNK- 005	Sample Name:B1		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8077	0.5536
Mean	2.18	2.18	0.8077	0.5536
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 006	Sample Name:B2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.16	2.16	0.8020	0.5623
Mean	2.16	2.16	0.8020	0.5623
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 007	Sample Name:B3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8090	0.5509
Mean	2.18	2.18	0.8090	0.5509
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 008	Sample Name:B4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.16	2.16	0.8028	0.5612

Mean	2.16	2.16	0.8028	0.5612
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK-009	Sample Name: C1		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8097	0.5497
Mean	2.18	2.18	0.8097	0.5497
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK-010	Sample Name: C2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8096	0.5511
Mean	2.18	2.18	0.8096	0.5511
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK-011	Sample Name:C3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8100	0.5466
Mean	2.18	2.18	0.3622	0.2366
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK-012	Sample Name:C4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.19	2.19	0.8124	0.5457
Mean	2.19	2.19	0.8124	0.5457
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK-013	Sample Name:D1		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.19	2.19	0.8141	0.5443
Mean	2.19	2.19	0.8141	0.5443
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK-014	Sample Name:D2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.19	2.19	0.8111	0.5443
Mean	2.19	2.19	0.8111	0.5443
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 015	Sample Name: D3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8075	0.5491
Mean	2.18	2.18	0.8075	0.5491
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 016	Sample Name: D4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8071	0.5574
Mean	2.18	2.18	0.8071	0.5574
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 017	Sample Name:X		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	24.70	24.70	0.5821	0.2855
Mean	24.70	24.70	0.5821	0.2855
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Detail Table 5/03/05 4:00 PM

Analysis Name : Fairene Leong
Comment : Zn (II) Adsorption , Time Effect and Loading Size
Meas. Date : 5/03/05 4:00 PM
Element : Zn

Sample ID	STD1	Sample Name	ABS	REF
No.	Corr. Conc (ppm)	Conc. (ppm)	0.0001	-0.0010
1	--	0.00	0.0001	-0.0010
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 2	Sample Name	ABS	REF
No	Corr. Conc (ppm)	Conc. (ppm)	0.0832	0.0166
1	--	0.20	0.0832	0.0166
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 3	Sample Name	ABS	REF
No	Corr. Conc	Conc. (ppm)	0.1581	0.0262
1	--	0.40	0.1581	0.0262
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 4	Sample Name	ABS	REF
No	Corr. Conc (ppm)	Conc. (ppm)	0.2968	0.0452
1	--	0.80	0.2968	0.0452
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Coefficient : K3=--
K2=--
K1=3.684285E-001
K0=5.600001E-003

Corr Coef : 0.9991

Sample ID	UNK- 001	Sample Name:D4/1hr		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	0.1329	0.1329	0.8141	0.5433
Mean	0.1329	0.1329	0.8141	0.5433
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 002	Sample Name: D4/2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.13	2.13	0.8002	0.5087
Mean	2.13	2.13	0.8002	0.5087
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 003	Sample Name: D4/3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	0.4652	0.4652	0.8321	0.5333
Mean	0.4652	0.4652	0.8321	0.5333
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 004	Sample Name:D4/4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	0.6492	0.6492	0.8700	0.4999
Mean	0.6492	0.6492	0.8700	0.4999
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00
	6.03			

Sample ID	UNK- 005	Sample Name:D4/0.2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	0.3655	0.3655	0.6999	0.4500
Mean	0.3655	0.3655	0.6999	0.4500
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 006	Sample Name:D4/0.3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	0.1994	0.1994	0.8003	0.5253
Mean	0.1994	0.1994	0.8003	0.5253
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 007	Sample Name:D4/0.4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	0.0033	0.0033	0.8013	0.3188
Mean	0.0033	0.0033	0.8013	0.3188
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 008	Sample Name:X		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF

1	24.70	24.70	0.8012	0.5121
Mean	24.70	24.70	0.8012	0.5121
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Detail Table 4/22/05 3:01 PM

Analysis Name : Fairene Leong
Comment : Cu(II) Adsorption , 2 hrs
Meas. Date : 4/22/05 3:01 PM
Element : Cu

Sample ID	STD1	Sample Name	ABS	REF
No.	Corr. Conc (ppm)	Conc. (ppm)	-0.0002	-0.0040
1	--	0.00	-0.0002	-0.0040
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--			

Sample ID	STD 2	Sample Name	ABS	REF
No	Corr. Conc (ppm)	Conc. (ppm)	0.0539	0.0345
1	--	1.00	0.0539	0.0345
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 3	Sample Name	ABS	REF
No	Corr. Conc	Conc. (ppm)	0.0878	0.0594
1	--	2.00	0.0878	0.0594
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 4	Sample Name	ABS	REF
No	Corr. Conc (ppm)	Conc. (ppm)	0.1809	0.1219
1	--	4.00	0.1809	0.1219
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Coefficient : K3=--
K2=--
K1=4.444571E-002
K0=2.820002E-002
Corr Coef : 0.9980

Sample ID	UNK- 001	Sample Name:A1		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	9.88	9.88	0.4183	0.2692
Mean	9.88	9.88	0.4183	0.2692
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 002	Sample Name:A2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	10.11	10.11	0.4152	0.2657
Mean	10.11	10.11	0.4152	0.2657
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 003	Sample Name:A3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	10.12	10.12	0.3941	0.2533
Mean	10.12	10.12	0.3941	0.2533
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 004	Sample Name:A4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	10.67	10.67	0.4322	0.2759
Mean	10.67	10.67	0.4322	0.2759
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 005	Sample Name:B1		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	13.03	13.03	0.5359	0.3354
Mean	13.03	13.03	0.5359	0.3354
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 006	Sample Name:B2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	11.94	11.94	0.5002	0.3140
Mean	11.94	11.94	0.5002	0.3140
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 007	Sample Name:B3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	12.33	12.33	0.4955	0.3124
Mean	12.33	12.33	0.4955	0.3124
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 008	Sample Name:B4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	11.86	11.86	0.4482	0.2867

Mean	11.86	11.86	0.4482	0.2867
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 009	Sample Name:C1		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	8.80	8.80	0.3184	0.2090
Mean	8.80	8.80	0.3184	0.2090
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 010	Sample Name:C2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	8.72	8.72	0.3751	0.2433
Mean	8.72	8.72	0.3751	0.2433
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 011	Sample Name:C3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	9.81	9.81	0.3622	0.2366
Mean	9.81	9.81	0.3622	0.2366
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 012	Sample Name:C4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	9.00	9.00	0.3276	0.2156
Mean	9.00	9.00	0.3276	0.2156
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 013	Sample Name:D1		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	6.83	6.83	0.2709	0.1809
Mean	6.83	6.83	0.2709	0.1809
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 014	Sample Name:D2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	8.14	8.14	0.3330	0.2184
Mean	8.14	8.14	0.3330	0.2184
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 015	Sample Name:D3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8075	0.5491
Mean	2.18	2.18	0.8075	0.5491
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 016	Sample Name:D4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.18	2.18	0.8071	0.5574
Mean	2.18	2.18	0.8071	0.5574
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 017	Sample Name: X		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	24.70	24.70	0.5821	0.2855
Mean	24.70	24.70	0.5821	0.2855
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Detail Table 5/03/05 3:00 PM

Analysis Name : Fairene Leong
Comment : Cu (II) Adsorption , Time Effect and Loading Size
Meas. Date : 5/03/05 3:00 PM
Element : Zn

Sample ID	STD1	Sample Name	ABS	REF
No.	Corr. Conc (ppm)	Conc. (ppm)	-0.0002	-0.0040
1	--	0.00	-0.0002	-0.0040
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 2	Sample Name	ABS	REF
No	Corr. Conc (ppm)	Conc. (ppm)	0.0540	0.0355
1	--	1.00	0.0540	0.0355
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 3	Sample Name	ABS	REF
No	Corr. Conc	Conc. (ppm)	0.0838	0.0604
1	--	2.00	0.0838	0.0604
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Sample ID	STD 4	Sample Name	ABS	REF
No	Corr. Conc (ppm)	Conc. (ppm)	0.1810	0.1216
1	--	4.00	0.1810	0.1216
Mean	--	--	0.0000	0.0000
SD	--	--	0.00	0.00
RSD(%)	--	--		

Coefficient : K3=--
K2=--
K1=4.434571E-002
K0=2.821002E-002
Corr Coef : 0.9970

Sample ID	UNK- 001	Sample Name:D4/1hr		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	17.31	17.31	0.4253	0.2892
Mean	17.31	17.31	0.4253	0.2892
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 002	Sample Name: D4/2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	6.83	6.83	0.4232	0.2887
Mean	6.83	6.83	0.4232	0.2887
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 003	Sample Name: D4/3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	17.81	17.81	0.3811	0.2333
Mean	17.81	17.81	0.3811	0.2333
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 004	Sample Name:D4/4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	6.03	6.03	0.4782	0.2709
Mean	6.03	6.03	0.4782	0.2709
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 005	Sample Name:D4/0.2		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	2.5862	2.5862	0.5229	0.3324
Mean	2.5862	2.5862	0.5229	0.3324
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 006	Sample Name:D4/0.3		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	0.5316	0.5316	0.5001	0.3240
Mean	0.5316	0.5316	0.5001	0.3240
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 007	Sample Name:D4/0.4		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF
1	0.0833	0.0833	0.4953	0.3126
Mean	0.0833	0.0833	0.4953	0.3126
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00

Sample ID	UNK- 008	Sample Name:X		
No	Corr. Conc (ppm)	Conc. (ppm)	ABS	REF

1	25.00	25.00	0.4472	0.2997
Mean	25.00	25.00	0.4472	0.2997
SD	0.00	0.00	0.0000	0.0000
RSD(%)	0.00	0.00	0.00	0.00